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THE INSTITUTE OF PETROLEUM

AN Ordinary General Meeting of the Institute of Petroleum was held at 26 Portland Place, London, W.1, on 13 April 1955, the Chair being taken by the President, Lt.-Col. S. J. M. Auld, O.B.E., M.C., D.Sc.

The Editor read the minutes of the previous meeting, which were confirmed and signed as a correct record. He also announced the names of members elected since the previous meeting, and gave notice of the date of the meeting.

The Chairman, introduced Mr J. B. Sandrock of Esso Development Co. Ltd., who read the following paper on behalf of the author.

REFINING METHODS FOR MAXIMIZING MIDDLE DISTILLATE PRODUCTION *

By L. S. BONNELL †

SUMMARY

PETROLEUM refiners, like many others producing consumer goods find it advantageous to make detailed studies of consumption trends. The Standard Oil Company (N.J.), affiliated and subsidiary companies (hereafter referred to as Jersey) have periodically studied overall market and refining trends. This has aided in planning refinery operations, and in deciding where additional refining and marketing facilities may be needed. In recent years, Jersey's studies generally indicated that the world-wide consumption of certain petroleum products will likely increase faster than for other products. Notably, the consumption of distillate fuels, compared with other products, has regularly increased in some areas, and this trend is expected to continue.

In the past, studies of refining operations in the U.S.A., made for Jersey by the Esso Research and Engineering Company (formerly Standard Oil Development Company), have shown that the problem of meeting the demand for distillate fuels can be solved largely by modifying available refining methods. This paper covers a brief, general investigation of the subject. It points out how refining schemes generally may be altered to increase distillate fuel production, with emphasis on the European refining area.

The results from the different refining schemes used in this study are intended to show approximate production potentials which might be realized in certain refining areas; namely, in Europe, the U.S.A., and Canada. For convenience, consideration will be confined to the total production of distillate fuels, termed middle distillate, and comprising kerosine, jet fuels, gas oil, and diesel fuels. Detailed studies beyond the scope of the present one would be needed, of course, to establish how specific refineries might increase middle distillate production economically.

INTRODUCTION

In some refining areas the forecast is that the distillate fuel consumption will probably increase faster than that for other major petroleum products. This is shown by recent studies of consumption and refinery production trends in three refining areas—Europe, the U.S.A., and Canada. In some areas the consumption trend for distillate fuels has been alarmingly sharp.

As a basis for this study, refining plans were set up for each of the three refining areas mentioned above. The base plan for each area reflected existing or immediate future average operations for several refineries in each area. In all cases, by changing the crude rate the volume of motor gasoline and speciality products—chiefly solvent naphthas, lubricating oils, and asphalts—were held constant as operations were modified to increase middle distillate production. Gasoline octane number was also kept constant. Several alternative refining plans were computed. In each, processing alterations were made to increase middle distillate production. Normally available modern refining equipment was assumed.

Generally speaking, refining operations are geared to meet the demand for motor gasoline and other major fuel products. In this circumstance, the ratio of middle distillate to motor gasoline is a useful yardstick. It readily measures in a simple way the effect of changing operations to increase middle distillate production when assuming that motor gasoline production must be held constant. With this in mind, a preliminary review was made of a number of different refining situations. Generally, a fairly large increase in middle distillate–motor gasoline ratio was indicated to be possible when (*a*) removing middle distillate from cracking feed stocks, (*b*) adding maximum virgin naphtha to middle distillate, and (*c*) increasing middle distillate final boiling point somewhat beyond the limit allowed in some areas by product specifications.

When applying the modifications (*a*)–(*c*) to the U.S.A. operations, the middle distillate–gasoline ratio could be increased from 1·06 to 1·9. To achieve this increase would require improved distillation and storage facilities. The crude rate had to be increased

* MS. received 2 March 1955.

† Esso Research and Engineering Company.

30 per cent in order to maintain constant volume of motor gasoline and speciality products.

In the European area, where refining operations are somewhat different from those in the U.S.A., it might be more difficult to increase the middle distillate–gasoline ratio. This would be largely because middle distillates for European sales do not include much cracked distillate. Also, the sulphur content of both bunker fuels and distillate fuels is a limiting factor. Notwithstanding, there are two steps which would improve middle distillate–gasoline ratio in Europe; namely, (*a*) remove virgin middle distillate fractions from cracking feed stock, and (*b*) remove such fractions from bunker fuel, assuming its sulphur specification could be met by using blending stocks that have been partially desulphurized. The overall effect of introducing these relatively simple changes in the refining plans studied, showed that middle distillate–motor gasoline ratio could be improved rather sharply, from a value of 0·75 for the base refining plan to 1·4.

A comparison of the European results with those from the U.S.A. study indicated how different market requirements and different crudes affect middle distillate production. The conclusion was that the middle distillate–motor gasoline ratio in Europe could be much improved by finding outlets for speciality products now included in motor gasoline. Also, this ratio could be raised by refining crudes giving relatively high middle distillate and low naphtha yields.

Analyses of the Canadian situation pointed to the problems facing the refiner marketing in a cold climate. Transportation problems also complicate the picture in Canada. This part of the study showed that middle distillate-gasoline ratio for the Canadian refineries could be varied over a wide range by widening the boiling range of the middle distillate fractions. It appeared possible that some of the advantages indicated by this step could be realized if adequate consumer and bulk storage facilities became available. Meeting critical pour specifications appeared to be the chief problem.

The conclusions from this study are based on rather general refining plans. These do not necessarily reflect the operations of any one refinery. Changes in operational factors, such as the type and amount of crude run, refining methods, and quality requirements, might alter the conclusions quantitatively. Economics certainly would be a deciding factor in most situations. In the final analysis, however, there remain two important ways of improving middle distillate supply:

1. Remove middle distillate from cracking feed stocks.
2. Widen the boiling range of middle distillate as far as quality restrictions permit.

Proper application of such steps should suffice to meet most of the immediate needs for increasing middle distillate volume. Thus, it should be expected that as requirements materialize, present refining methods will be more fully exploited wherever the economic incentive exists.

CONSUMPTION OUTLOOKS

Readily available information indicates that the consumption of middle distillate will certainly tend to increase faster than for other major petroleum products in the European refining area.[1] A convenient way of analysing this trend is to look at the ratio of middle distillate to motor gasoline which is shown in Table I. This compares the consumption ratio with the refinery output for the European industry. The 1953 figures are based on actual production and consumption data. On the surface, the

TABLE I

Estimated [1] Middle Distillate–Motor Gasoline Ratio

Year . . .	1953	1954	1955	1956
Consumption . .	1·05	1·06	1·07	1·08
Refinery production .	1·00	0·97	0·97	—

relatively small increase in middle distillate–motor gasoline ratio based on the estimated figures does not look too alarming. There does, however, appear to be a trend toward unbalance between supply and production ratios of these products. In addition, there is evidence of considerably greater unbalance of middle distillate supply and production in certain European countries. This raises the question of what steps might be taken in certain refineries to bring the middle distillate–motor gasoline ratios more nearly in balance.

In some other Jersey refining areas, notably in Canada, the trend in the middle distillate–motor gasoline ratio shown by product sales portends increasing shortages of middle distillate. Figures on this basis in Table II reveal that there was a sharp increase in this ratio for Imperial Oil Ltd. between 1949 and 1952. The Jersey world-wide figures also show a smaller but significant trend. In view of

TABLE II

Middle Distillate–Gasoline Ratios Based on Sales

Year . .	1949	1950	1952	1951	1953
Imperial Oil Ltd.[2]	0·62	0·70	0·73	0·73	0·74
Jersey world-wide [2] . .	0·72	0·77	0·79	0·80	0·77

these figures, supply problems are a possibility in certain refining areas.

BASES OF STUDY

A first step in this study was to set up base refining plans reflecting near-current operations. For any one area, the base plan corresponded to the average operations in a typical group of three or more refin-

eries. For example, the U.S.A. base-plan operations included deep vacuum crude distillation, fluid catalytic cracking, and thermal cracking of naphtha and crude pitch. Fig 1 is a greatly simplified process flow diagram showing the chief source of middle distillate and other major fuel products in the U.S.A. base case. A somewhat different plan was required to reflect European operations, as shown later.

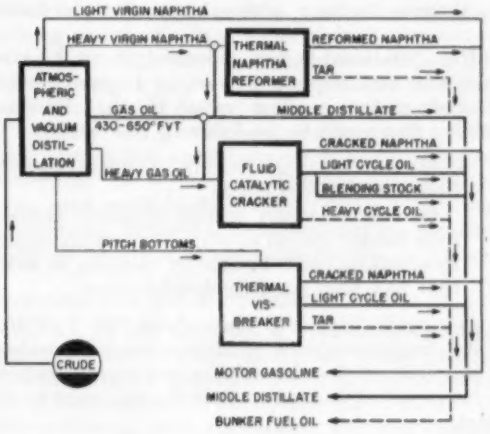

Fig 1

AVERAGE U.S.A. REFINING SCHEME

Explanatory: Only major processes are indicated. Minor processes were not taken into account except for thermal cracking of catalytic clarified oil and non-selective olefin polymerization. Middle distillate includes all distillate fuels—kerosine, diesel fuels, heating oil, and the like.

In computing the results for the base cases, certain approximations of minor importance were made for convenience. Another qualification is that only total middle distillate has been considered. No attempt was made to ascertain whether this distillate contained the required proportions of merchantable products. The requirements may vary widely from one location to another. Notwithstanding, past studies have shown that the total middle distillate will contain a sufficient variety of fractions so that ordinary consumption needs can be met. Considering all these qualifications, the results for any one refining plan are not necessarily typical of any refinery in a given area, but rather are intended to reflect a given general situation.

In the interest of realism, the base plans assumed the use of modern refining equipment already existing or about to be installed. The volume of all of the more important products were kept in line with anticipated consumption requirements, with this exception: the volume of bunker fuel oil was allowed to vary as processing was modified to increase middle distillate production. Also, gasoline octane number was held constant at near-current levels for each area, and the quality requirements of other major products were kept practically the same throughout. Sufficient raw materials for making so-called "speciality" products were set aside in order that their influence on middle distillate–motor gasoline ratio would be accounted for. The distinction made among major and speciality products is shown in Table III.

Table III

Major fuel products	Speciality products (constant volumes)
Motor gasoline (constant volume) *	Liquid petroleum gas
Middle distillate (variable volume)	Aviation gasoline
Bunker fuel oil (variable volume)	Solvent naphthas
	Lubricants and wax
	Asphalts and chemicals

* Constant octane number at level current in each area.

GENERAL EFFECTS OF REFINING METHODS

A preliminary review of certain refinery operations indicated that generally fairly large increases in middle distillate production could be realized by: (*a*) removing middle distillate from cracking feed stocks; (*b*) adding maximum virgin naphtha to middle distillate; and (*c*) increasing middle distillate end point somewhat beyond the limit allowed in some areas by product specifications. This was clearly shown by a detailed study of the U.S.A. situation.

Table IV

Summary of U.S.A. Refining Schemes

Case: / Characteristics:	1-D Base case	2-D No mid dist cracked	3-D Like 2-D with max naphtha in mid dist	4-D Like 3-D with higher FBP mid dist
Mid dist, FBP, ° F	620 (327° C)	620 (327° C)	620 (327° C)	700 (371° C)
% Virgin naphtha in mid dist	26·8	26·8	30·4	27·4
% Mid dist in cat cracking feed	28	0	0	0
Rates and Production 1000-b.c.d.				
Crude distillation	100	114	118	130
Motor gasoline	31·0	31·0	31·0	31·0
Middle distillate	32·7	44·5	48·2	58·9
Bunker fuel oil	13·1	16·1	16·1	16·6
Middle distillate–motor gasoline ratio	1·06	1·44	1·56	1·90

Table IV summarizes the results from the U.S.A. plans for refining average U.S.A. crude. A crude rate of 100,000 barrels/calendar day (b.c.d.) for the base case was assumed for convenience. In the following cases motor gasoline volume was held constant by increasing crude rate.

Case 2-D shows that by removing all distillate fuel fractions from cracking feed, an important boost in middle distillate volume would result. The increase in middle distillate–motor gasoline ratio to 1·44 from 1·06 was caused partly by the 14 per cent increase in crude rate needed to maintain a constant volume for all products, excepting middle distillate and bunker fuel.

Case 3-D shows the effect of raising the percentage of heavy naphtha included in middle distillate. This

could be achieved by improving the performance of crude fractionation facilities, without lowering distillate fuel flash specifications.

In Case 4-D middle distillate final boiling point (F.B.P.) was raised to 700° F (371° C) with a resultant large increase in distillate volume. This would involve a further improvement in fractionation facilities. Also, additional refining of heavy cracked distillates would be needed.

There were indications that most if not all of the principles found to apply in the U.S.A. refining plans could be used by refineries in other areas. With this background, the study was extended to the European and Canadian situations.

EUROPEAN OPERATIONS

Fig 2 is a simplified version of the base case showing the major processes and the sources of middle distillates for a group of European refineries for 1954. Aramco crude was assumed as fairly representative

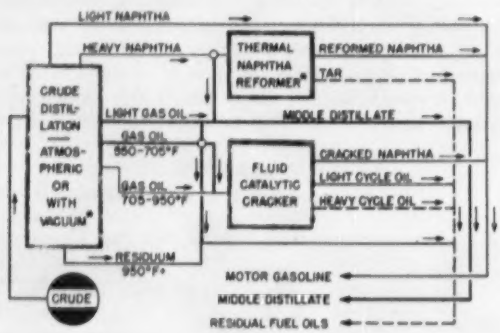

FIG 2

AVERAGE EUROPEAN REFINING SCHEME

Explanatory: Only major processes are indicated. Minor processes were not taken into account in computing European cases, except for non-selective olefin polymerization and treating of kerosine and lubricating oils. Middle distillate includes all distillate fuels—kerosine, jet and diesel fuels, and the like.

* If the refinery has a catalytic combination cracking unit, reformer vapour goes to combination unit tower and vacuum distillation is omitted.

for the average refinery. For convenience, the base case was assumed to be a full-scale refinery of a nominal capacity of 100,000 b.d. of crude distillation. Results for smaller refineries could readily be scaled down from these figures.

This base case reflected an average combination of various refinery operations; namely, atmospheric plus some vacuum distillation, fluid catalytic cracking, thermal naphtha reforming, and light olefin polmerization. The assumption was made that the middle distillate would comprise mainly crude fractions, since most European distillate fuels contain no cracked materials. Middle distillate end point was carried to about 705° F (374° C) final boiling point (F.B.P.). This F.B.P. was considered to be limited by the sulphur content of the major middle distillate fractions. Another factor in European operations, not important in the U.S.A. picture, is that sulphur content of bunker fuel normally is a serious problem. Thus, to approach the sulphur specifications, some virgin (crude) middle distillate must be added to bunker fuels in excess of that needed to meet the usual specifications of gravity, or viscosity, or both. Finally, because there is a ready residual fuel market, no thermal cracking (visbreaking) of crude residua is ordinarily used.

The operational factors described above indicated that the two steps which might improve middle distillate–motor gasoline ratio in the European refining area would be the following:

(*a*) remove virgin middle distillate fractions from cracking feed stocks;

(*b*) remove virgin middle distillate fractions from bunker fuel oil assuming the bunker sulphur specification could be met by blending in stocks that had been partially desulphurized.

A case producing higher than about 705° F (374° C) F.B.P. fractions in middle distillate was not considered a practical one because sulphur and sometimes pour-point specification tend to limit the end point to this level.

Table V summarizes the principal results for the European cases discussed below.

TABLE V

Summary of European Refining Schemes

Case Number:	1-E (Base)	2-E	3-E	4*
% Virgin naphtha in mid dist (max possible)	40	36	35	30
% Mid dist in cracking feed	41	0	0	0
Limiting bunker fuel spec	Sulphur	Sulphur	Viscosity	Viscosity
Rates, 1000 b.c.d.:				
Crude distillation	100	130	130	105
Cat cracking, fresh feed	18·3	14·5	14·5	15·4
Production, 1000 b.c.d.:				
Motor gasoline	32·4	32·4	32·4	32·4
Middle distillate	24·2	43·4	45·8	42·0
Bunker fuel oil	26·2	34·1	31·8	13·8
Virgin mid dist included in fuel oil	(3·7)	(6·0)	(3·6)	(0)
Middle distillate–motor gasoline ratio	0·75	1·34	1·41	1·30
Bunker fuel oil quality:				
°API gravity	14·2	14·9	13·6	11·5
Sp. gr.	0·9712	0·9665	0·9752	0·9895
S.F. vis at 122° F	60	80	150	150
% Sulphur	3·4	3·4	3·5	4·1

* U.S.-type operations assumed. See text.

Base Case 1-*E*. As already stated, the base case reflects average, approximate 1954 results, which were anticipated for the average European refining situation. This case showed a 0·75 middle distillate–motor gasoline ratio, well below the 1·08 ratio predicted for internal European consumption in 1956.

Case 2-*E*. This case shows the effect of removing all 435° to 705° F (224° to 374° C) boiling range middle distillate from cracking feeds. As expected, the result was a large increase in middle distillate production over the preceding case, with the middle distillate–motor gasoline ratio increased from 0·75 to 1·34. This simple step alone, therefore, brought the ratio to a

considerably higher figure than the 1·08 consumption ratio estimated for 1956.

Case 2-*E* assumes provision of adequate distillation and storage facilities for producing and segregating all middle distillate fractions. Since bunker fuel sulphur influences the amount of middle distillate needed in the blend, this case was set up to give bunker fuel oil of the same sulphur content as computed for the base case. A greatly increased crude rate was needed to offset loss in gasoline yield incurred by not cracking middle distillate materials.

Case 3-*E*. This case, a modification of Case 2-E, shows the effect of limiting the amount of middle distillate in bunker fuel oil to the extent needed only by viscosity and gravity specifications. The result was a further but small increase in middle distillate production. It is possible that the fuel blending stocks used could be hydrofined to reduce their sulphur content, thereby allowing sulphur specifications on fuel oil blends to be met more readily.

Comparison with U.S.A. Operations

Case 4. In this case, U.S.A.-type operations were applied to the average European crude, making European product distribution and quality excepting bunker fuel sulphur. That is, referring to Fig 1, deep vacuum flashing, pitch visbreaking, and fluid catalytic cracking of maximum available gas oil (excluding middle distillate) was assumed. The chief purpose of this case was to illustrate the effect of minimizing bunker fuel oil as accomplished in U.S.A. operations.

Case 4 shows a 1·30 ratio (middle distillate–motor gasoline) as compared with 1·41 for Case 3-E because of the additional processing by catalytic cracking. From this it seems apparent that the type of processing in the European cases was not responsible for the lower ratio.

Further, comparing the U.S.A. and European results, the final U.S.A. case showed a middle distillate–motor gasoline ratio of 1·9, compared to the 1·3-ratio shown in Case 4. This raised the question of what causes contributed to this marked difference in ratio. Analysis indicated that the 1·9 ratio could be achieved in Europe when allowing for the following factors :

(*a*) In the U.S.A. cases, compared to Europe, motor gasoline yield on crude is about 5 per cent lower because some potential motor gasoline fractions are sold as aviation gasoline, solvents, and chemicals.

(*b*) Sale of 2·4 per cent on crude of fuel oil blending stock in the European cases. This product is really middle distillate, but is not considered as such.

(*c*) The Aramco crude refined in Europe contains 6 per cent more virgin gasoline without a compensating increase in middle distillate content, compared to average U.S.A. crude.

The ratio (middle distillate–motor gasoline) in Europe could be much improved by finding outlets for speciality products now included in motor gasoline. Also, it would help to discontinue making fuel oil products requiring excessive amounts of middle distillate fractions in order to meet sulphur specifications.

In brief, the study of the European cases indicated that immediate future needs for middle distillates can probably be approached by refraining from cracking middle distillate fractions. To accomplish this would require improved fractionation and storage facilities.

CANADIAN OPERATIONS

The results of studies on the Canadian operations are of interest here, since they demonstrate principles applicable to the European problem. The situation in Canada can be likened to that which would face refineries supplying the market in the colder countries, Norway and Sweden, for example.

The high middle distillate consumption in Canada stems chiefly from two basic causes :

(1) Canada's cold climate naturally creates a high demand for distillate fuels.

(2) There are no all-the-year-round warm areas consuming gasoline which can readily supply distillate fuels to those having high distillate demand. Other factors complicate the problem of meeting this demand. Pour and cloud point specifications, important because of the cold climate, definitely limit middle distillate production.

The situation facing some of the Canadian refineries was examined in some detail with the aid of a base case, typical of 1953 operations, and some possible future refining schemes. These were set up to represent an average of three plants, typical of refineries operating in different Canadian provinces. The objective was to determine what could be done to increase middle distillate supplies by widening the boiling range of the middle distillate fractions, since in the Canadian operations the amount of middle distillate included in cracking feed stocks had already been reduced to a practical minimum. The other characteristics of the Canadian operations are similar to those for the U.S. refineries shown in Fig 1.

TABLE VI

Summary of Canadian Refining Schemes

Case Number:	1-C (Base)	2-C	3-C	4-C
Approx characteristics of mid dist :				
Flash, ° F	140	115	115	115
Boiling range, ° F (nominal)	350–675	300–700	300–725	280–725
Boiling range, ° C (nominal)	177–357	149–371	149–385	138–385
Pour, ° F	−10	−10	0	0
Relative crude rate and major products, 1000 b.c.d. :				
Crude run	100·0	106·7	111·0	118·2
Motor gasoline	37·2	37·2	37·2	37·2
Middle distillate	30·6	36·6	39·1	46·2
Ratio :				
Middle distillate–motor gasoline	0·82	0·98	1·05	1·24

Referring first to Case 1-C (base) a crude rate of 100,000 b.c.d. was assumed for convenience. A middle distillate–gasoline ratio of 0·82 was obtained.

Comparing Case 2-C with 1-C, lowering the middle distillate average flash by 25° F (−4° C) would permit including additional heavy naphtha fractions in middle distillate and raising the final boiling point. This would increase the ratio of middle distillate to gasoline from 0·82 (base) to 0·98, an important improvement. Case 3-C shows a further possible increase (to 1·08 from 0·98) in the middle distillate–gasoline ratio as a result of raising limiting pour point by 10° F (−12° C). This small increase is obtained by increasing final boiling point. Because of the difficulty of segregating low- and high-pour stocks in transit and in consumer storage tanks, this case offers little promise. However, Case 3-C shows the magnitude of possible gain if it became possible to segregate distillates having, say, −10° F (−12° C) and +0° F (−18° C) pour points. This possibly could be achieved with segregated storage for special low-pour products made in small volume for the colder marketing areas.

Case 4-C shows a possible important increase in the ratio up to 1·24 if all naphtha fractions down to 280° F (138° C) initial boiling point could be included in middle distillate. Superfractionation would be necessary to include such a large quantity of naphtha fractions while maintaining flash specifications. This procedure, while technically feasible, would probably be fairly costly.

The above comparisons serve chiefly to point out that the middle distillate–motor gasoline ratio can be varied over a wide range if average middle distillate boiling range can be widened without exceeding critical product quality requirements. Such requirements, as this study indicated, are sometimes unduly restricted because adequate consumer and bulk storage facilities are not available.

PROCESS DEVELOPMENT TO IMPROVE MIDDLE DISTILLATE YIELDS

The foregoing comparisons are limited to specific applications of established processes to specific refining situations. It is of interest to go somewhat beyond these comparisons and review whether modifications of existing processes might improve middle distillate production. Also, some speculation is appropriate on how newer processes under development might influence the picture.

Gas Oil Conversion Processes

The thought has been expressed that what is needed to supply more middle distillates is a conversion process that would result in a high middle distillate–gasoline ratio. So far as known, there is no good process for accomplishing this end. At present the most common conversion processes used for upgrading the less saleable crude fractions do not result in a high middle distillate–motor gasoline ratio. For catalytic cracking, the major process, this ratio is about 0·5, taking naphtha from polymerization into account. Without light olefin polmerization, the ratio would be about 0·65. Thermal gas oil cracking would give a similar result.

It can be visualized that a selective cracking process to produce principally middle distillates from heavy residual fractions would be a very desirable solution of the supply problem. But it is improbable that such a process could be developed easily, considering our current knowledge of the hydrocarbon structures present in the heavy oils, the known strengths of the carbon–carbon bonds in these structures, and the mechanisms by which cracking takes place. Cracking thermally and with well-known cracking catalysts produces middle distillate and motor gasoline in a surprisingly near-constant ratio.

Application of relatively inexpensive catalytic cracking produces high octane gasoline throughout the entire C_4–430° F (221° C) boiling range permissible in motor gasoline. The value of this high octane material is much higher in gasoline than its value in middle distillate. Therefore, a conversion process for making middle distillate rather than gasoline could not compete with catalytic cracking to make high quality gasoline, unless it was considerably lower in operating cost than catalytic cracking. It is not impossible that such a process could be developed; it is, however, likely to be difficult.

Residuum Conversion Processes

Past refining studies have shown that there is little gain in middle distillate production when using known refining methods for reducing fuel oil production. This is illustrated in Table VII comparing refining cases for U.S.A.-type operations. Motor gasoline production and octane number were held constant, and middle distillate production was maximized. Vacuum-flashed pitch (7·5 per cent on crude) was processed in turn by thermal visbreaking, by de-asphalting, and by coking. U.S.A. average crude was assumed.

Table VII

Pitch processing method	Thermal vis-breaking	De-as-phalting	Coking
Crude rate, 1000 b.c.d.	97	91	89
Major products, 1000 b.c.d.:			
Motor gasoline	33·9	33·9	33·9
Middle distillate	37·5	36·7	39·7
Ratio: Mid Dist–Motor Gas	1·11	1·08	1·17
Bunker fuel oil	8·7	3·8	0

Coking slightly improved middle distillate production, but the change in middle distillate–gasoline ratio from the first case to the last was quite small. Much the same result would be obtained for the Aramco crude used in the European cases. But if this same type of comparison were made starting with a much heavier (lower °API gravity) crude, coking would result in a greater increase in middle distillate–gasoline ratio

over the visbreaking case. This is because heavy crudes require a larger amount of middle distillate fractions in bunker fuel to meet viscosity requirements than medium or light crudes. Coking releases such fractions for other uses.

Visbreaking of residuum fractions obtained by either atmospheric or vacuum distillation ordinarily gives about a 1 : 1 middle distillate–gasoline ratio. However, the distillate volume from visbreaking is usually small, and the quality of the gasoline is poor.

Fluid Coking

The results shown above for the coking case are fairly representative for the average U.S.A. refinery using fluid coking. This new Jersey coking process, which has recently reached commercial use in the U.S.A., appears to have definite advantages over the older coking processes. It gives a higher yield of useful products and a somewhat lower coke yield, and the investment required is significantly lower. From the viewpoint of the refiner, fluid coking has definite promise in locations where a reduction in residual fuel production is needed. Speciality uses for the granular coke produced may aid in making the process economically attractive.[4]

Pour Point Reduction

One approach to increasing middle distillate yield would be to find ways of reducing the effect which high-pour components have upon the pour and cloud points of a blend. It might be possible, for example, to develop additives that reduce cloud and pour points.

Influence of Crude Quality

It is obvious, perhaps, that refining crudes containing relatively large amounts of middle distillate fractions will result in high middle distillate–motor gasoline ratios. Crudes containing large amounts of heavy gas oil and residual fractions require large amounts of cracking, which lowers the ratio. For this reason, those refineries having need for a high distillate fuel–gasoline ratio should process the lighter crudes when possible. The exact relationship between crude distillation characteristics and middle distillate–gasoline ratio has not been established in this study. However, it should bear close examination for those refineries where a choice of crude can be made.

Hydroforming

Hydroforming, a process for making high octane gasoline from low grade naphthas, will generally have little or no effect on middle distillate–motor gasoline ratio. This statement assumes use of the process in a conventional refinery having catalytic cracking. When hydroforming is substituted for thermal reforming in such a refinery, keeping total motor gasoline octane number constant, the ratio (middle distillate–motor gasoline) would decrease by about 3 to 4 per cent. This occurs because for a given octane number level, hydroforming increases gasoline yield. On the other hand, when hydroforming is substituted for thermal reforming to permit making a higher octane gasoline pool, the ratio (middle distillate to motor gasoline) may be essentially unchanged.

In a refining situation where modern cracking facilities do not exist and a market for very high amounts of residual fuels is available, hydroforming would conceivably find application. A higher middle distillate–motor gasoline ratio would be possible generally under this condition compared to that obtained where motor gasoline quality and yield come largely from catalytic cracking.

Hydrodesulphurization and Hydrofining

Hydrodesulphurization, a modified catalytic hydrogenation process for sulphur reduction, is being installed in some of the European and other refineries where high-sulphur stocks are a problem. It might find application particularly in making diesel oils from high-sulphur stocks, provided the hydrogen required for the process is not excessive. This would permit materials high in sulphur to be upgraded from bunker fuel blending agents to middle distillates. In addition, it may allow smaller amounts of middle distillate fractions to be used as bunker fuel blending agents in cases where sulphur is a limiting specification on bunker fuel.

Hydrofining, a modification of hydrodesulphurization, is used for improving the stability of certain cracked fractions, thereby permitting their inclusion in distillate fuels. This process, since it gives only a small reduction in sulphur, would not accomplish the benefits of hydrodesulphurization.

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DISCUSSION

C. V. Hill: The subject of Mr Bonnell's paper is of vital interest to European refiners. As Mr Bonnell points out, the paper is of necessity based on averages and broad principles, and the question arises as to how far the assumptions which are essential to calculations can be related to the practice in any one refinery.

Two of the assumptions made by Mr Bonnell appear to present possible difficulties when applied to individual refineries. One is that the total middle distillate can readily be split into marketable products. I can visualize the case of a refinery where there is a very large demand for kerosine, with the result that all the very low sulphur material would be taken out of the bulk middle distillate. The remainder might well be of too high a sulphur content for diesel engine road vehicle fuel, and might therefore have to be confined to marine diesel fuel. If that refinery

did not have an adequate off-take for bunkers there might be some expensive cross-hauls from one refinery to another, in order that that surplus marine diesel fuel could be disposed of, while an importation of gas oil of suitable sulphur content for the actual proper market of that refinery might also be required. This indicates the hydrofining of some of the distillate not for fuel oil, but for the middle distillate itself.

The other assumption which worries me a little is that, in some cases, the whole of the fuel oil of a given refinery can be disposed of at ships' bunkers. In my experience, this is seldom the case, and a high proportion of the fuel oil taken from any one refinery must be of a considerably lower viscosity than those in Table V for 150 Saybolt Furol at 122° F. If any refinery could dispose of the whole of its fuel oil at Bunker C viscosity, it would be the new refinery at Aden, which is situated at one of the biggest bunkering ports in the world. Nevertheless, only two-thirds of the fuel made at the Aden refinery can be disposed of at as high a viscosity as 150 Saybolt Furol, *i.e.* 3000 seconds Red. I at 100° F. The remainder is required at 60 Furol or below.

I had a calculation made for one particular refinery which meets Mr Bonnell's conditions for average European operation, *i.e.* for distillation, reforming, and fluid cat cracking, operating 50 per cent of its throughput on high sulphur Middle East crude, and the remainder on one of the lower sulphur crudes, so that the overall crude input can therefore be regarded as a fair average of Middle East production. When Mr Bonnell refers to the removal of all the 435° to 705° F boiling range from the cracking feeds, I assume he does not imply scientifically perfect, but good fractionation within the limits of the ordinary refinery distillation unit. By following Mr Bonnell's line of thought a middle distillate–motor gasoline ratio of 1·34 can be achieved on paper, and this is almost exactly in line with 2E, Table V. The resulting bunker fuel oil is not 80 sec Furol as in Mr Bonnell's Table, but 800, *i.e.* 20,000 sec Red. I at 100° F. Now, in order to reduce the viscosity to what is considered to be marketable, namely 150 Furol, I have used catalytically cracked gas oil as a cutter stock, because that is the highest sulphur, and therefore probably the least desirable component of the middle distillate. When that has been done and fuel cut back to give 3000 sec Red. I at 100° F, the middle distillate–motor gasoline ratio falls at once, from 1·34 to 1·08, and even at this ratio the whole of the refinery make of fuel oil is at 3000 sec viscosity. The pattern of the market demand in this particular refinery is such that it will absorb less than half the make at this high viscosity, the remainder being at 1000, 650, and 250 sec Red. I at 100° F. The effect of bringing the viscosity of the fuel oil down to the customers' demand limit is to bring the middle distillate–motor gasoline ratio below one.

As a result of these rather interesting calculations, I am unable to decide to what extent the line of attack which was adopted by Mr Bonnell is, in fact, applicable in practice at one refinery, as opposed to a group of average refineries. I would like to ask Mr Sandrock whether he considers that any one European refinery will be able to do better than a ratio of one, without introducing these new processes to which Mr Bonnell referred, desulphurization in particular.

J. B. Sandrock: The paper states that one should not consider any one refinery as a specific problem, but rather should one consider a large area, and therefore it is extremely difficult, except in certain very particular circumstances, to apply it to a given refinery. No two refineries are the same. What one has, another lacks, and vice versa, and in general, one has to consider an area situation, so that what one gains on the swings, one loses on the round-abouts.

I did not check Mr Hill's calculations on the high viscosity fuel oil, so I do not know the reason for the result. Possibly Mr Bonnell's European cases were based on Aramco crude entirely, and that might have some effect on the high viscosity factor.

In connexion with the first point that Mr Hill made, here again he says that single refineries may, for example, have large kerosine requirements. I would like to emphasize again that this should not be applied to single refineries. I would say, if that is the case, and one wants a low sulphur kerosine, and not a high sulphur diesel fuel, it is necessary to install a hydrofining unit.

J. H. G. Carlile: Mr Bonnell's paper is a timely reminder that the present shortage of middle distillate fuels constitutes a very real problem. Whether this problem will be more acute in the future is a matter on which we can only speculate, but there are indications that substantially increased demands for diesel fuel and jet fuel will have to be met. On the marine side there has been a marked increase in the use of diesel engines since 1948, and it has been estimated that by 1958 motorships will account for some 40 per cent of world tonnage. Diesel rail traction is also becoming well established in various parts of the world, and the recent plan for modernization of British Railways proposes a large increase in the use of diesel locomotives in the U.K.

I was rather surprised to note the base case selected for U.S. refining schemes (Table IV) was one in which the middle distillate–motor gasoline ratio was as high as 1·06. If this was in fact representative of average U.S. refining, which in 1953 gave a ratio of the order of 0·55, there would be no problem of a shortage of middle distillate fuels but rather the reverse. Similarly, whereas average European refining gives a middle distillate–motor gasoline ratio of approximately 1 (Table I) a lower ratio is used in the base case quoted in Table V. Would the same potential improvements of middle distillate–motor gasoline ratio which are shown to be possible for selected cases apply to the average ones?

J. B. Sandrock: On the U.S. basic case question, those figures are specifically for the average of three typical U.S. refineries, on which the base case is built up. They do not necessarily reflect operations throughout the U.S.A. In fact, if one averaged the results of all the U.S. refineries it might not necessarily come to that. I think Mr Bonnell selected refineries with a view to covering as wide a range as possible of U.S. practice, but the actual figures may vary a bit if one takes into consideration a few more refineries or a specific refinery.

In the case of the European query, there is the base case here with a ratio of 0·75. That also was obtained by averaging a number of specific refineries. The figures given in the paper of refinery production were taken, I think, from an O.E.E.C., publication, and they relate to the whole of Europe. There again, it is possible that there would be some discrepancies. I agree that in this case it appears to be fairly wide. The figure given in Table V for the European base case is an average for a given number of specific refineries, whereas the figure given on page 274 for refinery production is one which was taken from an O.E.E.C., source, not one of our own, and there may have been a slightly different basis for compiling the figures, so the figure for the whole of Europe may vary a little from the European base case.

G. M. Barrett: Mr Bonnell mentioned in his paper that by extending the gas oil boiling range one would lower the Flash Point by about 25° F. I would like to ask whether any consideration was given to the possible increase in vapour pressure if one drops the Flash Point by putting more volatiles in this material, and whether that is likely to cause any trouble in diesel engine injection systems, and whether there is any particular test which could be applied and which could control that property.

J. B. Sandrock: I should say in this case, no. It referred to one of the Canadian examples, and one ascertained by how much this could increase the ratio, the result would show that other properties of the fuel were not, in this case, taken into account.

R. L. Cowley: Mr Hill mentioned the distribution of the various products within the middle distillates. Is it not true that the percentage increase in diesel fuel consumption in Europe is greater than that of the total middle distillates, and has this been taken into account when these figures were arrived at?

J. B. Sandrock: No, that point was not especially taken into account. As mentioned previously, Mr Bonnell lumped all the middle distillates together. It was his feeling that over an area these things tend to average out. It may be that in specific countries or in specific situations, one has a larger demand for a particular cut of a middle distillate, than in another. His paper states, that he has had to make that assumption. Naturally, it is necessary to make quite a few assumptions. One of these, *i.e.* lumping all the middle distillates together, for purposes of simplification; otherwise, one has to cope with rather a large number of cases if one tries splitting it down further.

Wm. P. Given: In many of the countries concerned there is an artificial retail price for diesel fuel. Within the oil industry, the tendency is to make the price f.o.b. so that the refinery can eventually meet the law of supply and demand. In many countries diesel fuel for road use does not carry such a heavy tax as that for gasoline. To that extent, an artificial factor is introduced in the natural flow of the commodities. Likewise, to the extent that a gasoline tax represents a luxury tax, rather than a tax for road use and so forth, the economic balance is being shifted and should, at least, be compensated for by an equivalent tax on diesel fuel.

The other thought that arises is that in California asphalt is being burnt in boilers. I realize that can be done best in a stationary application, but it will help to raise the overall viscosity of fuel sold, and also in the future when the heavy demand for fuel is expected to lessen. Another ratio, which is equally important here, is the distillate fuel–heavy fuel oil ratio—making a three-cornered analysis desirable. In the same connexion there is the use of low-grade gasoline for heating purposes—about which we shall hear more in the near future. I understand that in France a small space-heater is available, which will burn heavy straight-run gasoline catalytically without any great danger.

R. C. Porter: I was a little disappointed, if I may say so, that Mr Bonnell in his paper did not forecast more of the technical developments in the new processes which may synthesize, either from the top or the bottom, additional middle distillates. It seems to me that, in the long run, if this trend continues, the solution might lie in one of those directions, and I was wondering whether anybody could forecast how soon and from which end the synthetic distillates are likely to develop.

J. B. Sandrock: Mr Bonnell makes the point that we would all like to see a middle distillate processed from heavy fuel oil. Unfortunately, molecules at present do not always go in the direction that one wants them to, and it would obviously be a little difficult to make one thing and not get some other things. The processes which have been and are being developed at present on converting fuel oil, do in fact, at the same time make gasoline, and when one makes gasoline as well as middle distillate, one is still not getting the ratio exactly the way one wants it. It is a little difficult to predict or speculate on what process will be developed next week or next year. Naturally, quite a few people are looking into this matter.

Dr G. B. Maxwell: Those of us who have to handle the Middle East crudes are constantly up against this question of sulphur content, particularly in regard to high-speed diesel fuel. That may explain one of the additional reasons why the ratio of gas oil–gasoline in the U.S.A. is mostly rather better than in the U.K. Generally the U.S.A. handles lower sulphur crudes, and for that reason, it can cut more deeply into crude to produce virgin gas oil and also includes more thermal and catalytically cracked gas oil in both high-speed diesel and in heating oils. European refineries at the present time are generally confined to high-speed diesel fuel with a limit of about 1·0 per cent of sulphur. Most of the specifications run to about that. During the second world war, the limit was raised in many areas up to about 1·25, and outside certain restrictive equipment, and also under certain conditions, such as in fishing fleets, where one has long periods at low loads and low jacket temperatures, there seemed to be no adverse effects (very few were reported) from raising the sulphur content of high-speed diesel from 1·0 to 1·25. Currently, in some markets the U.K. is now being asked to produce high-speed diesel fuel with a sulphur content as low as 0·4, and from Middle East crudes the only way of coping with this problem is to desulphurize. Are we allowing ourselves as refiners to be pushed in a line, merely from competition, which is cutting our own throats as regards the yields of the middle distillates we are going to get? That is one of the points which perhaps Mr Sandrock may care to amplify.

Similarly, though to a lesser extent, there is the question of flash point. In the U.S.A., much of the gas oil that is produced contains material which in the U.K. would normally be included in a kerosine fraction. However, one cannot lengthen one's kerosine–gas oil cuts at the expense of gasoline, as much as one would like, without coming up against the flash point limitation.

J. B. Sandrock: From a technical standpoint, most people are agreed that these 0·4 sulphur specifications for automotive diesel fuel are restrictive, and that were it not for a competitive situation, one probably would not consider going as low as that. I, personally, find that it is unjustified, but one has to consider the marketers who sell the stuff, and eventually, I suppose, pay us, and they seem to like these lower levels.

In connexion with the flash point, in some circumstances this is restrictive and if it could be relaxed a little, it would certainly assist in making more middle distillate and improving one's ratio. Both these points are very important, but I am not sure whether, as technical people, it is possible to do very much about them.

W. M. Catchpole: Mr Sandrock in his final remark, really brought out a point that Mr Bonnell made in his paper, on the question of pour point. Even if desulphurization comes along for competitive reasons, one will still be left with the problem of the wax which is naturally present in Middle East crudes, and particularly in the case of road vehicles and rail-road transport, pour point is an important consideration. One cannot leave the fuel to solidify in the vehicle tank during cold weather. Mr Bonnell has stated that what one really needs in order to provide a large increase in middle distillates is a cheap process for removing wax, or an additive which will modify wax crystallization, which is a method that I do not think will be economic. Failing these, one must try to develop a process which will control the type of molecule; *i. e.* when cracking residue, one must somehow control the breakdown so that less gas and gasoline is made than in the conventional processes. I see no other simple way of making more middle distillate at the expense of gasoline.

In Table VII I presume all the figures apply to the same crude, and I would like to ask whether the coking figures are based on the actual commercial operations from the one fluid coker that I assume is operating, or are they based on predictions from small-scale work?

J. B. Sandrock: These figures are all based on the same crude, on what Mr Bonnell calls the U.S. average crude, which he assumes throughout his study. The figures on coking are not based on commercial operations, because the paper was actually written before the first fluid coker, which is now operating very successfully, had started up. They are based on pilot plant work, and are therefore the estimated yield for a commercial operation.

Lt.-Col. S. J. M. Auld: I have the very pleasant task of congratulating Mr Bonnell on his admirable paper, and on his choice of friend and colleague who has spoken for him. Mr Sandrock has discussed specifications, synthesis, sulphur removal, and many other considerations, and his efficient handling of Mr Bonnell's paper is very much appreciated.

The vote of thanks was accorded with acclamation.

CONTINUOUSLY RECORDING OXIDATION APPARATUS FOR HYDROCARBON OILS*

By D. G. CHILDS.

SUMMARY

An all-glass apparatus which will record continuously the oxygen uptake of an oxidizing oil sample has been developed, and its method of operation and use described.

Oxygen is circulated continuously through the oxidizing sample at constant pressure and temperature, while volume decrements are recorded automatically.

By a suitable choice of soluble catalyst concentration, the apparatus has been used successfully both for assessing the inherent oxidation stability of straight insulating oils and for comparing the performances of various anti-oxidants in insulating oil.

INTRODUCTION

WORK on insulating oils in the past has shown that for the purposes of quality control where the desired properties of a commercial product are defined in a specification, oxidation tests of fixed duration are useful. The tests usually consist of comparing the extent of formation of sludge and/or acid with a specified maximum after a test period of rigidly defined oxidizing conditions. However, if a new product is to be investigated, or a study of anti-oxidants to be made, some means of obtaining a continuous record of the course of oxidation of an oil sample is valuable. Part of the work on insulating oils in the Central Electricity Research Laboratories concerns the use of anti-oxidants, which function by delaying the onset of serious oxidation in an oil by a time known as the "induction period." Successful use has been made of this induction period both here and elsewhere [1] as a means of comparing inhibited oils using apparatus in which the induction period in oxygen absorption is determined under accelerated conditions. Measurement of this time by itself, however, gives only an indication of the efficacy of the anti-oxidant and of the inhibitor response of the oil. It gives no information about the inherent oxidation stability of the base oil before treatment on any influence spent anti-oxidant may have on the course of the oxidation after the induction period. The apparatus described in the present paper was developed to obtain information on these points and to provide a test, complementary to the pressure oxidation test,[2] that would differentiate between uninhibited oils of different oxidation stability.

Oxygen absorption lends itself well to continuous recording and is the first step in any oxidation process. It has been used by many workers in the past, both on insulating oils,[1, 3, 4, 5, 6, 7] and pure hydrocarbons,[8, 9, 10, 11, 12] and recently correlation between the induction period in oxygen absorption in a laboratory test and the actual service performance of an insulating oil has been reported.[13]

The present paper describes a new apparatus which records continuously the oxygen uptake of an oxidizing sample, and some of the results which have been obtained from its use on insulating oils. The apparatus is fully automatic, fairly compact and has an all-glass gas system.

BRIEF DESCRIPTION OF APPARATUS

The continuously-recording oxidation apparatus is essentially a modified Dornte [8] apparatus. Oxygen is circulated continuously through the oil which is held at constant temperature in an oil bath. Decrements of pressure in the gas system, due to oxidation of the sample are made good by the upward movements of dibutyl phthalate in a gas burette which is arranged to maintain a constant average pressure. The changes in the liquid level in the gas burette with time are recorded automatically, and consequently, the apparatus plots changes in volume at constant pressure.

DETAILED DESCRIPTION OF APPARATUS

General

The apparatus, which is constructed entirely of Pyrex glass with standard ground-glass joints sealed with silicone high-vacuum grease, is shown diagrammatically in Fig 1. Figs 2 and 3 are photographs of the front and rear views of the assembly. The circuits of the ancillary electronic equipment are shown in Figs 4 and 5.

Circulatory System

The sample under test, together with any catalyst which may be added, is contained in a 250-ml conical flask (N, Fig 1), maintained at constant temperature in an oil bath which can be lowered to facilitate the removal of the reaction flask. The Liebig condenser (L), returns volatile oxidation products to the sample. An all-glass, double-acting, magnetic pump with glass ball valves (P), passes oxygen through a bubbler (Q)

* MS received 13 May 1955.

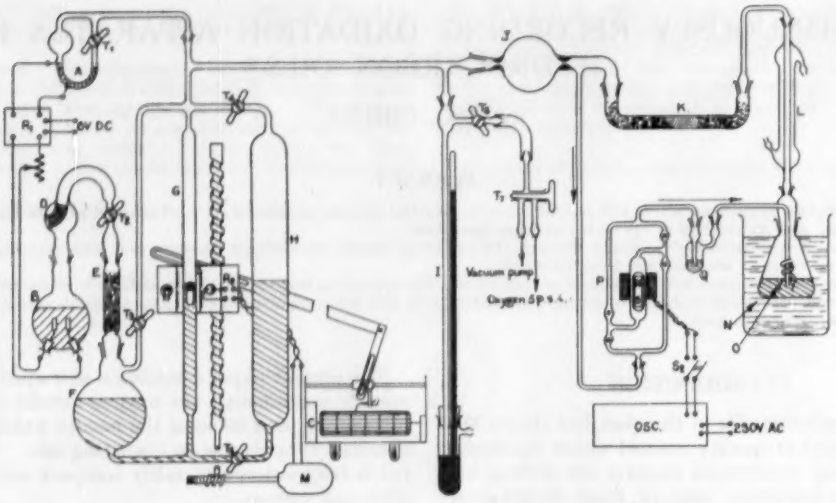

FIG 1

CONTINUOUSLY RECORDING OXIDATION APPARATUS

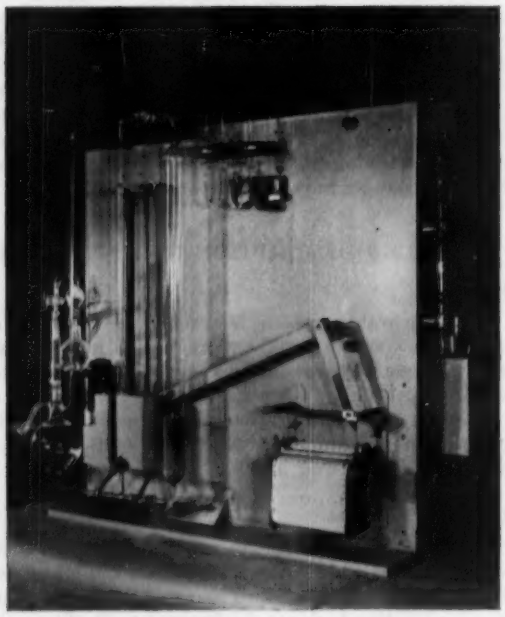

FIG 2

CONTINUOUSLY RECORDING OXIDATION APPARATUS
(Front view)

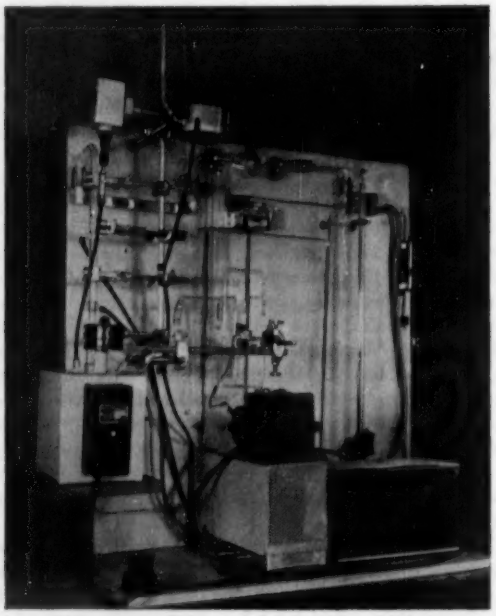

FIG 3

CONTINUOUSLY RECORDING OXIDATION APPARATUS
(Rear view)

and then into the sample through a "gas lift disperser" (O). The bubbles of oxygen from the central delivery tube pass up the spiral outer tube carrying oil with them. This allows longer oxygen–oil contact, stirs the oil, and reduces diffusion effects. The gas then passes through the condenser to an absorption tube (K), containing silica gel backed with Anhydrone

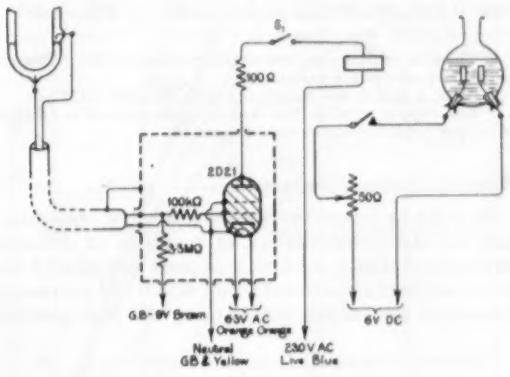

FIG 4

CIRCUIT OF MANOSTAT RELAY R1

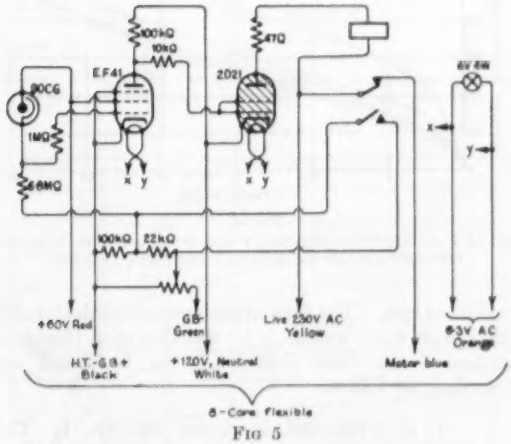

FIG 5

CIRCUIT OF PHOTO-ELECTRIC FOLLOWER R2

(magnesium perchlorate) for the removal of water, and soda asbestos (Carbosorb) backed with Anhydrone for the absorption of carbon dioxide. The oxygen is subsequently returned to the pump for recirculation.

Manostat Constant Pressure System

The manostat used in the apparatus is based on that described by Bolland,[14] and operates in the following manner.

Initially, the apparatus is filled with oxygen to the working pressure and taps T1, T3, and T6 of Fig 1 are closed. Taps T2, T4, and T5 are open. The manometer switch (A) contains ethylene glycol saturated with potassium chloride, and this electrolyte is balanced between the reference pressure of the gas contained between it and tap T1 on the one side and the pressure of the gas in the rest of the apparatus on the other. Any decrease in pressure in the circulatory system, which is connected to the manostat system through a pressure-wave damping tube (J), results in a movement of the electrolyte in the manostat and the consequent operation of relay R1. The latter controls the electrolysis of a saturated aqueous solution of oxalic acid in flask B, from which the carbon dioxide and hydrogen generated pass through a mist trap (D), and a silica gel drying tube (E), to the gas burette reservoir (F). The increase in pressure in the reservoir forces the dibutyl phthalate which it contains into the limbs of the gas burette (G and H), restoring the pressure of the circulatory system to its average value and causing the contacts of the manostat, and thus relay R1, to open. This cycle of events is repeated continuously until the reservoir is empty and the gas burette full, which corresponds to a recorded volume of approximately 1 litre.

Recording Gas Burette

The changes in the level of the dibutyl phthalate in the gas burette are followed photo-electrically. An opaque float in the narrower limb (G), interrupts a beam of light from a bulb mounted on a trolley which can move vertically along the length of the gas burette. Upward movements of the float allow the light beam to fall on to the cathode of a gas-filled photo-electric cell which controls relay R2. A slow-speed motor (M), operated by relay R2 drives the trolley upwards by means of a lead screw until the light beam is interrupted by the float, and the follower returns to its quiescent condition.

A micro-switch, not shown in the figure, is so arranged that all 230-V supplies to the apparatus are disconnected when the trolley reaches the end of its traverse. When it is desired that the follower should remain inoperative, a removable cover may be placed over the light beam. The trolley drive mechanism is provided with a release so that it may be moved along its guides independently of the lead screw. The movements of the trolley are transmitted mechanically to a sliding pen-arm, writing on a chart driven by an electric clock (C). The ratio of this transmission is variable and allows the apparatus to be adjusted so that the full scale of the 6-inch chart corresponds to a change in level in the gas burette equivalent to 1 litre.

OPERATING DETAILS

Starting the Apparatus

(*a*) Weigh 100 g of the oil sample to be tested with any catalyst which may be required into a clean dry 250-ml B24 conical flask, and connect this to the apparatus.

(*b*) Raise the oil bath into position.

(*c*) Open the apparatus to the atmosphere by turning tap T7 to the pump line with the pump disconnected, and opening tap T6.

(*d*) Open taps T1, T2, T3, T4, and T5.

(*e*) Close tap T2.

(*f*) Connect the vacuum pump to T7.

(*g*) Evacuate the apparatus, and when evacuation is complete, turn tap T7 to fill the apparatus with oxygen to atmospheric pressure.

(*h*) Repeat step (*g*).

(*i*) Repeat step (*g*).

(*j*) Open tap T2.

(*k*) Readjust the pressure of oxygen in the apparatus to atmospheric, if necessary.

(*l*) Close taps T1, T3, and T6.

(*m*) Switch on the manostat (S1, Fig 4).

(*n*) Remove the cover from the light beam on the follower.

(*o*) Adjust the pen-arm to zero on the chart.

(*p*) Adjust the clock to the correct time.

(*q*) Switch on the pump (S2, Fig 1).

(*r*) Note the time of starting on the chart.

Stopping the Apparatus

(*a*) Switch off the pump (S2, Fig 1).

(*b*) Cover the light beam of the follower.

(*c*) Lower the follower trolley until the pen-arm is at zero on the chart.

(*d*) Switch off the manostat (S1, Fig 4).

(*e*) Open tap T1.

(*f*) Carefully open tap T3 so that the liquid levels in the gas burette and reservoir slowly equalize, without drawing gas past the float.

(*g*) Lower the oil bath.

(*h*) Remove the reaction flask from the apparatus.

(*i*) Close tap T2.

(*j*) Remove the gas disperser tube and the Liebig condenser and wash both parts with chloroform followed by hot sulphuric acid–chromic acid mixture. After rinsing with copious hot water and distilled water dry in a hot air oven.

(*k*) Replace the cleaned parts and seal the apparatus with a clean dry 250-ml conical flask.

RESULTS OF INVESTIGATIONS

Effect of Pumping Rate

In order to determine whether the rate of oxidation of the sample under test would be limited by the rate at which oxygen could be supplied to the oil by the pump, tests were made in which the oxygen pumping rate was varied. The results of these tests are shown in Table I. These show that above 2·5 c.p.s., an increase in pumping rate has no effect on the rate of oxidation of even a Class A oil containing a high catalyst concentration. Accordingly, a pumping rate of 2·75 c.p.s. was chosen as standard for the rest of the work described.

TABLE I

Sample under test	Oxygen absorption rate ml/100 g/hr at pumping rate * of:		
	2·0 c.p.s.	2·5 c.p.s.	3·0 c.p.s.
Class A † oil with 100 p.p.m. Cu ‡	85	112	112
Class B † oil with 100 p.p.m. Cu ‡	85	107	107

* The rate of pumping was noted in terms of the frequency of the magnetic pump in c.p.s.

† Class A and B oils complying with BS 148 : 1933.

‡ The copper catalyst was added in the form of a solution of copper naphthenate in transformer oil.

Effect of Catalyst Concentration

In order to choose the optimum catalyst concentration for the differentiation of base oils of different oxidation stability, a number of tests was carried out with a series of experimental oils, which had previously been used for similar work [4] using the high-pressure

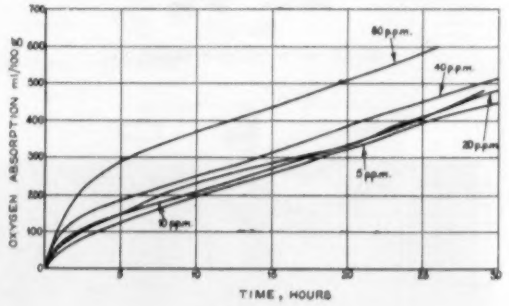

FIG 6

OXYGEN ABSORPTION CURVES OF EIO NO. 1 WITH INCREASING CONCENTRATION OF SOLUBLE COPPER CATALYST

oxidation test. The four experimental insulating oils (EIO) were made available by the Thornton Research Centre of the Shell Petroleum Co. Ltd. and are described as follows:

(1) *Experimental insulating oil No.* 1. The solvent refined feedstock from which transformer oils are normally produced by further oleum and earth treatment.

(2) *Experimental insulating oil No.* 4. A normal type of transformer oil.

(3) *Experimental insulating oil No.* 5. A normal type of transformer oil of higher aromatic content than No. 4.

(4) *Experimental insulating oil No.* 6. A technical white oil produced by heavy oleum treatment of a solvent refined stock.

The oils were oxidized in the apparatus, each with a series of increasing catalyst concentrations at a test temperature of 120° C. The catalyst used throughout these experiments was copper naphthenate containing

6·8 per cent copper, which was added in the form of a concentrated solution in transformer oil to give a resulting concentration in the test sample which has been expressed as parts of copper per million parts of oil. The families of oxygen absorption curves obtained from these tests are shown in Figs 6, 7, 8, and 9.

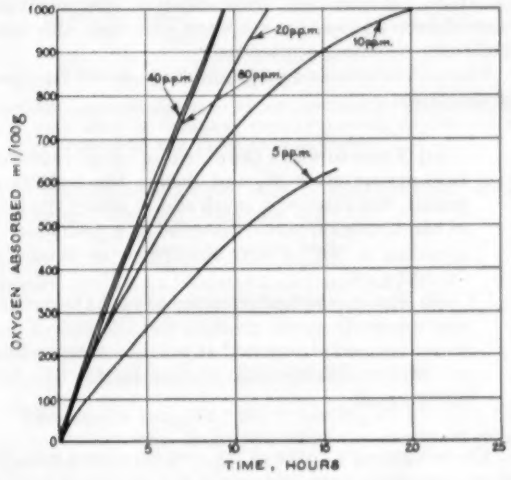

FIG 7

OXYGEN ABSORPTION CURVES OF EIO NO. 4 WITH INCREASING CONCENTRATION OF SOLUBLE COPPER CATALYST

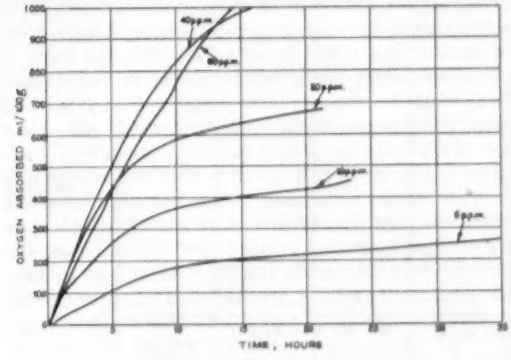

FIG 8

OXYGEN ABSORPTION CURVES OF EIO NO. 5 WITH INCREASING CONCENTRATION OF SOLUBLE COPPER CATALYST

Effect of Degree of Refinement

By comparing the recordings from the experiments described in the previous section for any one catalyst concentration, the effect of the base oil on the shape of the oxygen absorption curve obtained may be seen. Figs 10, 11, and 12 compare the traces obtained with the various experimental oils with catalyst concentrations of 5, 10, and 20 p.p.m.

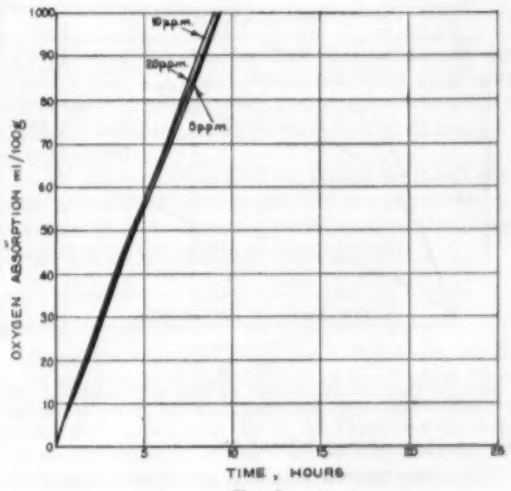

FIG 9

OXYGEN ABSORPTION CURVES OF EIO NO. 6 WITH INCREASING CONCENTRATION OF SOLUBLE COPPER CATALYST

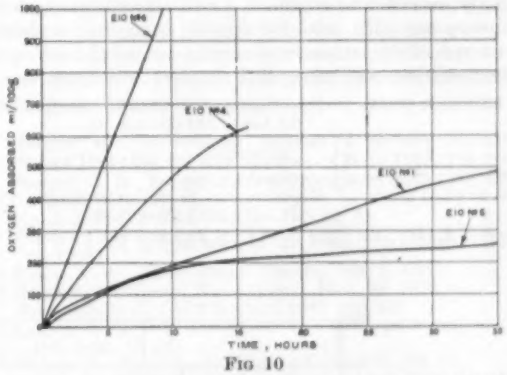

FIG 10

OXYGEN ABSORPTION CURVES OF THE EXPERIMENTAL OILS WITH A CATALYST CONCENTRATION OF 5 P.P.M. COPPER

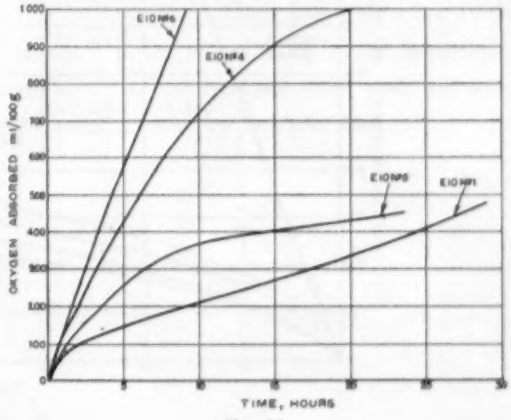

FIG 11

OXYGEN ABSORPTION CURVES OF THE EXPERIMENTAL OILS WITH A CATALYST CONCENTRATION OF 10 P.P.M. COPPER

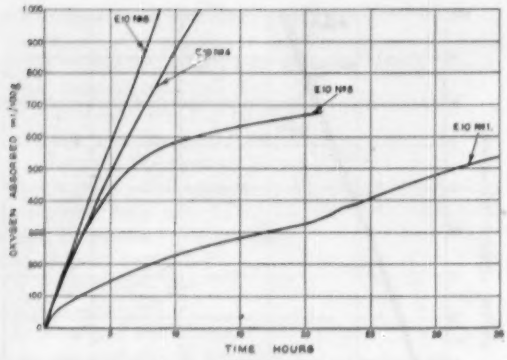

FIG 12

OXYGEN ABSORPTION CURVES OF THE EXPERIMENTAL OILS WITH A CATALYST CONCENTRATION OF 20 P.P.M. COPPER

Comparison of Uninhibited Oils

From the figures described in the foregoing section it can be seen that the best differentiation between base oils of different stability was obtained with a catalyst concentration of 5 p.p.m. copper. Accordingly, in order to establish a basis for comparison for future use with oils of unknown oxidation stability, a series of oxidations was carried out on the following transformer oils, using this catalyst concentration.

A.30 oil A (1944) . Experimental oil refined to comply with BS 148 : 1933 Class A.30.

BS 148 : 1951 oil A, BS 148 : 1951 oil B, BS 148 : 1951 oil C } Normal transformer oils refined to comply with BS 148 : 1951 by various manufacturers.

B.30 oil D (1944) . Experimental oil refined to comply with BS 148 : 1933 Class B.30.

The results obtained are shown in Fig 13.

The Testing of Inhibited Oils

Former work on inhibited oils using the static oxygen-absorption method for the measurement of induction period both by Beaven *et al* [1] and by the author has shown that at a test temperature of 120° C, a catalyst concentration of 100 p.p.m. of copper is very suitable for providing a comparative test of short duration. Accordingly, this catalyst concentration was chosen for testing inhibited oils with the continuously-recording apparatus.

The curves obtained with inhibited oils fell into two main classes :

(*a*) Those in which the extent of oxygen absorption at the start of, and during the induction period, was relatively small and in which the end of the induction period occurred at a point corresponding to an oxygen absorption of about 50 ml/100 g oil.

(*b*) Those in which the initial oxygen absorption was relatively much greater, and the end of the induction period occurred at points corresponding to oxygen absorptions in the range 100–200 ml/100 g oil.

The oils giving a curve of the first type were usually those containing anti-oxidants of the secondary amine type, while those oils giving curves in the second category contained anti-oxidants of the hindered phenol type.

Fig 14 shows this effect clearly. The oxygen absorption curves are those of the same base oil, alone,

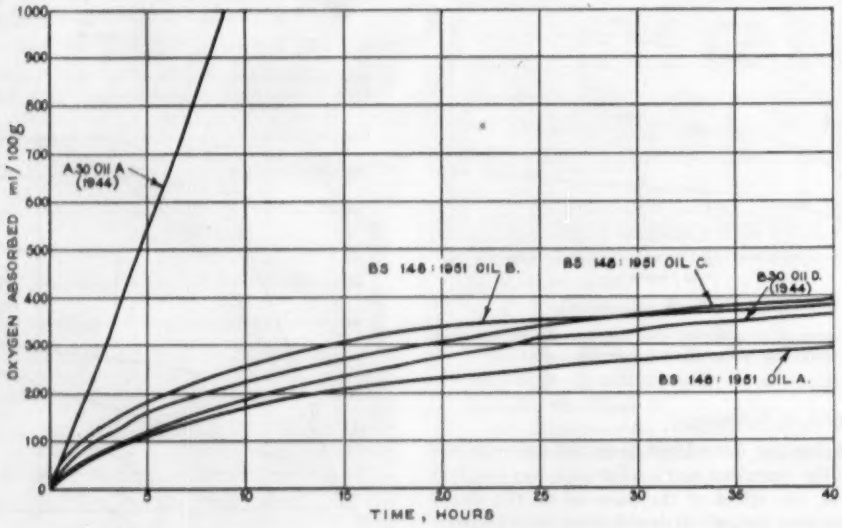

FIG 13

OXYGEN ABSORPTION CURVES OF TRANSFORMER OILS WITH A CATALYST CONCENTRATION OF 5 P.P.M. COPPER

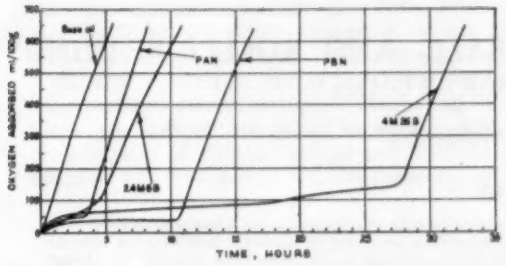

FIG 14

OXYGEN ABSORPTION CURVES OF INHIBITED TRANSFORMER OILS WITH A CATALYST CONCENTRATION OF 100 P.P.M. COPPER (0·25 PER CENT ANTI-OXIDANT)

and containing 0·25 per cent of four different anti-oxidants. The anti-oxidants are :

PAN . . Phenyl-α-naphthylamine.
PBN . . Phenyl-β-naphthylamine.
24M6B . . 2,4-Dimethyl-6-*tert*-butyl-phenol.
4M26B . . 4-Methyl-2,6-*ditert*-butyl-phenol (D.B.P.C.).

OBSERVATIONS ON RESULTS

The results indicate that in testing an oil with the continuously-recording apparatus, the choice of catalyst concentration depends on the nature of the oil under test. For oils containing no additives, and for which an indication of the inherent oxidation stability is required, a low catalyst concentration around 5 p.p.m. copper, is most suitable. In the case of oils containing anti-oxidants, where the oxidation must be sufficiently accelerated to provide a test of reasonably short duration, a higher catalyst concentration is more suitable. A value of 100 p.p.m. copper has been found by means of the static oxidation method to be sufficiently higher than the critical value above which the induction period is not appreciably shortened, and gives induction periods of up to 30 hr with this apparatus. Since many induction period values had been obtained with this catalyst concentration it was adopted for the work described. All the inhibited oils tested in this way showed induction periods, and none gave a greater oxygen absorption rate after the induction period than the rate given by the corresponding untreated base oil.

CONCLUSIONS

By choosing the catalyst concentration to suit the oil under test, the continuously-recording apparatus may be used for assessing either the oxidation stability of non-inhibited oil or the performance of oil containing anti-oxidant.

Under the conditions used for testing inhibited oils, spent anti-oxidant did not give rise to oxygen absorption rates after the induction period greater than those obtained with the corresponding base oils.

ACKNOWLEDGMENTS

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EVALUATION OF GREASES FOR BALL- AND ROLLER-BEARINGS AT LOW TEMPERATURES*

By J. W. PEARSON†

SUMMARY

An apparatus has been developed that may be used for measurements at constant shear stress or constant rate of shear on grease in bearings at low temperatures. Measurements of starting, breakaway, and running torques may be made. The apparatus employs a liquid cold bath for rapid cooling, and provision is made for thrust loading to minimize skidding of the balls of the test bearing, so reducing some anomalies in results observed hitherto with other low-temperature torque equipment.

For the interpretation of measurements made with the new apparatus a method is proposed which promises to reduce the variations in assessed low-temperature properties due to considerable differences in test bearings. However, a method of interpretation already in use gives equally good results if the test bearings are specially selected.

INTRODUCTION

Aircraft have had to meet low-temperature conditions of ever-increasing severity in developing all-weather operation, and Arctic and high altitude flying; failures have often been attributed to the use of unsuitable greases. Hence, low-temperature requirements and tests were introduced into the relevant specifications of aviation greases. The tests included penetration measurements at low temperatures [1, 2] and the shearing of the greases in a ball and socket apparatus,[3] but both types of test were rather unsatisfactory. In 1942–43 the U.S. services introduced a low-temperature test in which the test piece was a ball-bearing of the type commonly used in aircraft;[4] with minor modifications the test still appears in current U.S. specifications.[5] A simplified form of the apparatus, employing the same test bearings, was adopted in 1944 by the British authorities [6] and this test also appears in current specifications.[7]

Work at "Shell" laboratories showed that the British apparatus and test procedure could produce some anomalous results. Unexpectedly low results sometimes observed were thought to be associated with "skidding" of the balls of the bearing on the tracks, *i.e.* instead of the mainly rolling motion normally occurring there was much more sliding than usual. This view was supported by visual observation of the relative motion of the cage and races under the test conditions.

No cognizance was taken of skidding by the U.S. or the British services' low-temperature torque tests, but the Co-ordinating Research Council (CRC) [8, 9] and the joint committee of the Annular Bearing Engineers Committee/National Lubricating Grease Institute (ABEC/NLGI) [10] recognized skidding as a possible contributor to variability of results, and they advocated the use of a 5-lb thrust pre-load on the test bearing to reduce this effect. The British apparatus was adaptable to the application of a radial load for reducing the tendency to skid, but not, without major modifications, to the more desirable thrust loading. (In a ball-journal bearing subjected solely to thrust load each ball will carry an equal load, but since the axes of the ball loads are offset to the axis of the applied load the average ball loading is greater than under the same radial load; hence, there is less possibility of skidding under a thrust load than under the same radial load.)

Moreover, the British apparatus was not readily adaptable for measurements at a constant rate of shear (in contrast to measurements at a constant shear stress as in the British and U.S. grease specification tests); a constant rate of shear is a better basis for assessing torque requirements for grease-lubricated bearings in aircraft.[11, 12] A further disadvantage of the British apparatus had been found by the IP Mechanical Tests of Greases Panel, who showed that test bearings, even when procured from one manufacturer, could give widely different results for the time of one revolution when using a given grease under standard conditions.

Since the British apparatus was not adaptable, a critical review was made of commonly used equipment for determining the low-temperature characteristics of greases. The object was to select the most satisfactory apparatus or to design an apparatus incorporating the best features of the existing equipment. As a result of the survey it was decided to design a new apparatus to satisfy the following criteria:

(1) The apparatus should employ only the test bearing most commonly used in such tests, namely a ball bearing of 20 mm bore, 47 mm overall diameter, and 14 mm width (BRL 020). This restriction would simplify the construction and operation of the apparatus.

(2) The design should allow of thrust-loading the test bearing with a definite load transmitted directly to the bearing in order to reduce skidding. It should be possible to change the load during a test.

(3) The test bearing temperature should be measured by thermocouples.

(4) The cooling medium should be a liquid, thus increasing the rate of heat transfer and reducing

* MS received 20 August 1954.

† "Shell" Research Limited, Thornton Research Centre.

the time required for cooling. Use of the existing cold bath of the DTD apparatus would be advantageous and should be attempted.

(5) The apparatus should be suitable for measurements at constant shear stress or at constant rate of shear, and should be adapted to the measurement of starting torques and breakaway torques [11, 13, 14] as well as running torques.

EXPERIMENTAL

Fig 1 shows diagrammatically the lay-out of the apparatus. The test bearing is mounted at the bottom of a vertical spindle supported at the top by a parallel

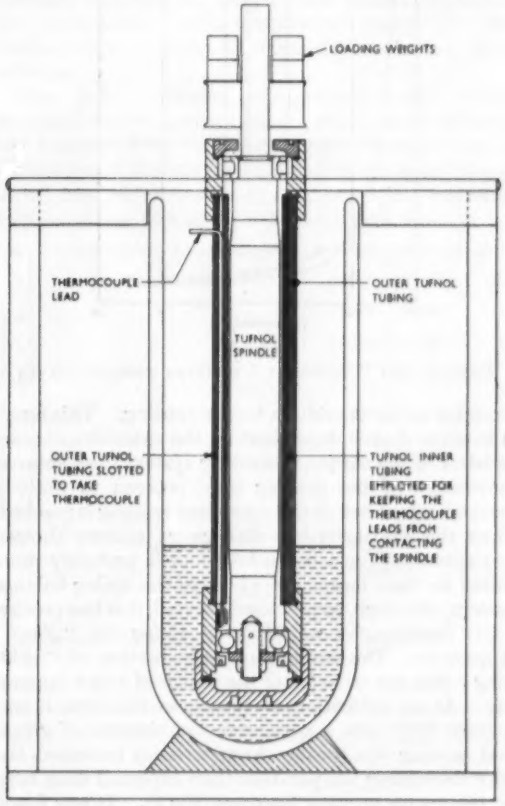
LOADING WEIGHTS
THERMOCOUPLE LEAD
OUTER TUFNOL TUBING
TUFNOL SPINDLE
OUTER TUFNOL TUBING SLOTTED TO TAKE THERMOCOUPLE
TUFNOL INNER TUBING EMPLOYED FOR KEEPING THE THERMOCOUPLE LEADS FROM CONTACTING THE SPINDLE

FIG 1

"SHELL" LOW-TEMPERATURE TORQUE APPARATUS FOR GREASE TESTING

roller bearing without lips on the outer race. Thus, the test bearing may be thrust-loaded by applying weights to the top of the spindle, and this load, which may be readily varied during tests, is transmitted without appreciable loss through the roller bearing direct to the test bearing. This method of loading is restricted to a maximum of about 30 lb. For higher loads a slightly modified top was designed for the spindle, which then uses spring loading. The modified top, Fig 2, is easily attached to the normal apparatus and may be used for loads up to about 1000 lb. The lower end of the housing is closed by a liquid-tight cover. The dimensions are such that the DTD cold-bath may be used. The mean bearing temperature is

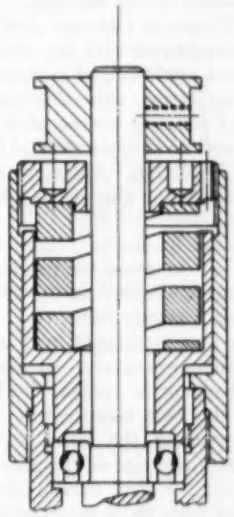

FIG 2

DIAGRAM OF MODIFIED TOP FOR SPRING LOADING

measured by two thermo-couples connected in series, one of which is spring-loaded against the outer race of the bearing while the other floats in the air gap just above the cage of the bearing. A close approximation to the actual grease temperature should thus be obtained.

For measurements under constant shearing stress, the torque is applied by a cord, wrapped round the spindle pulley, which passes over an idler pulley mounted on the cabinet surrounding the cold bath. The required load is attached to the end of the cord.

For measurements at a constant rate of shear the cord from the spindle pulley is passed over a movable pulley assembly, which includes a spring balance, to a constant-speed motor. An alternative procedure in which the spring balance was replaced by a cantilever bar, the deflection of which was measured by a clock indicator, was tried but measurements were not so accurate owing to hysteresis in the deflection of the bar. The use of strain gauges in conjunction with the cantilever bar was considered, but such complications in a measuring apparatus would outweigh the advantages.

PROCEDURES AND RESULTS

(a) *Measurements at Constant Shear Stress.* Preliminary work was directed to the comparison of results using the "Shell" apparatus, without added thrust load, with those using the British DTD apparatus, under conditions of constant shearing stress at an applied torque of 2000 g.cm. The detailed procedure followed that given in DTD specifications using the

DTD apparatus, *e.g.* DTD 577. As may be seen from Table I the "Shell" apparatus gave results lower than the DTD apparatus, and there was no indication that the "Shell" apparatus reduced the effect on the test results of differences in test bearings.

(b) *Running Torque at Constant Rate of Shear.* In view of earlier experience with the British apparatus and the published criticisms of measurements under constant shearing stress, attention was directed to measurements of running torque at a constant rate of shear. The method of measurement finally adopted is given in the Appendix. Again, most of the work reported was done in the absence of additional thrust loads.

It may be noted in the procedure that steps are taken to "run in" at room temperature in order to reduce the amount of rotation, at the test temperature, required to reach a steady indication of the running torque. This is also facilitated by "winding back" between each test temperature. It may also be seen that the test bearing is cooled slowly but is not maintained at the test temperature for prolonged periods, thus reducing the time required to take measurements over a range of temperatures. It was shown experimentally that results obtained in this way did not differ from those recorded after maintaining the bearing at the test temperature for periods up to 2 hr.

TABLE I

Comparisons of Results with British Specification (DTD) and "Shell" Apparatuses at Constant Torque (2000 g.cm)

Grease	Bearing No.	Mean time of revolution, sec			
		British (DTD) apparatus		"Shell" apparatus	
		Test 1	Test 2	Test 1	Test 2
A	1	30·5	34·3	22·3	21
	2	23·3	17·5	11·8	10·3
	3	15·8	14·2	8·4	—
B	1	7·4	6·0	3·7	2·9
	2	3·4	4·0	1·5	1·2

The types of curves obtained, which are plotted as a compound function of torque and speed of rotation (torque-rate ratio) against temperature, are shown in Fig 3.

(c) *Starting Torque and Breakaway Torque.* As arranged for measurements at constant rate of shear the apparatus may also be used for the determination of starting and breakaway torques. Starting torque is defined as the torque required just to initiate movement of the bearing, while breakaway torque is the torque required to accelerate the bearing to a given speed of rotation. These measurements may be made in a variety of ways. Thus, a bearing may be packed with grease at room temperature and cooled to the test temperature without prior rotation. Alternatively, the packed bearing may be "run in" at room temperature prior to cooling; this procedure should give results which are more repeatable but it may be less related to service conditions. In either case, the bearing is not moved during cooling and the motor is switched on when the test temperature is reached. The starting torque is noted when the spindle just moves and the breakaway torque, at the speed of rotation used, is

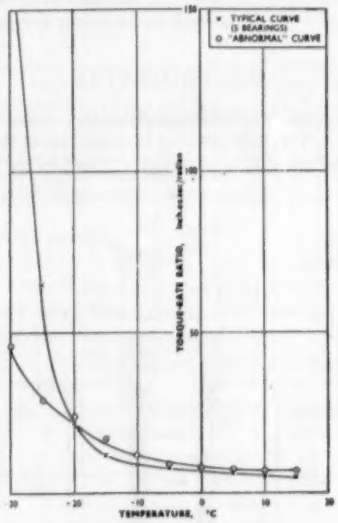

FIG 3

TYPICAL AND "ABNORMAL" RUNNING TORQUE CURVES

recorded as the maximum torque reading. This figure is to some degree dependent on the extension characteristics of the torque measuring system used because movement of the bearing may proceed to quite a marked extent before the maximum reading is reached. Thus the cantilever-bar–dial gauge system showed less extension for a given load and is probably more suited to these measurements than the spring balance system, although, as previously stated, it is less precise.

(d) *Investigation of Skidding Using the "Shell" Apparatus.* The elimination or reduction of "skidding" was one criterion of the design of a new apparatus. As an indication of success in this aim, it was noticed that with a particular combination of grease and bearing the torque characteristics increased less with decreasing temperature than expected from tests on the grease in other bearings (Fig 3). It was found that loading the test bearing by suitable weights placed on the spindle gave results more in line with those from the other bearings. This grease–bearing combination was therefore examined by mounting the apparatus in a cold chamber in such a way that the test bearing could be observed. The movement of the cage corresponding to one revolution of the inner race was measured at different temperatures, the rate of rotation of the spindle being 2 r.p.m., with the results in Table II. When the bearing was loaded with a weight of 15 lb the cage movement relative to the inner race was normal, showing that the skidding which occurred in the unloaded state had been

TABLE II

Visual Observations on Skidding with One Grease and Bearing

Temperature	Cage movement per revolution of inner race in 32nds of a revolution (Theoretical value : 12·5)
+15° C	Slightly >12
−15° C	Slightly >12
−20° C	10
−25° C	9
−30° C	8
−30° C *	Slightly >12

* Bearing loaded to about 15 lb.

The bearing used was fitted with a machined cage centred on the shoulders of the inner race and contained eight balls.

virtually eliminated. Under these loaded conditions the torque-rate ratio–temperature curve for the bearing approximated to those obtained using other bearings.

Figs 4–10 represent torque-rate ratio curves measured on two greases using a set of seven different test bearings from the same source. Examination of the shapes of the curves obtained without additional thrust load suggests that in only two cases was skidding occurring to a great extent. These were in the two tests with bearing 3 on Grease 2. When 10-lb load was applied to the test bearing the curves approximated more closely to those obtained with the other bearings, suggesting that skidding had been reduced. It is interesting to note that, of the series, bearing 3 showed the greatest diametric slackness, a character-

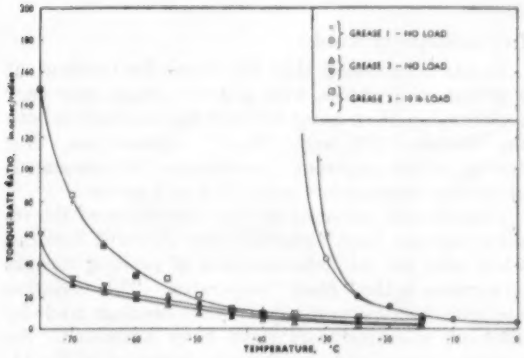

FIG 4

EFFECT OF TEMPERATURE ON RUNNING TORQUE

Bearing 3

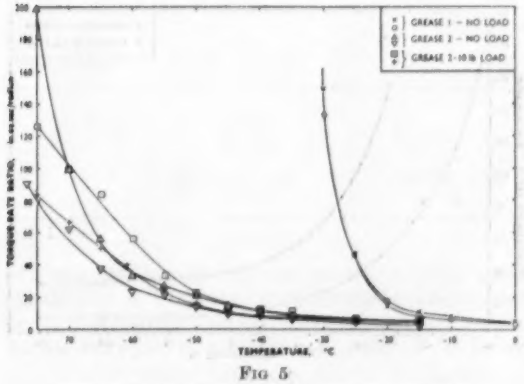

FIG 5

EFFECT OF TEMPERATURE ON RUNNING TORQUE

Bearing 19

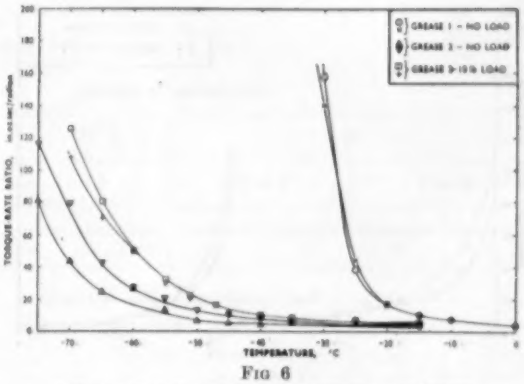

FIG 6

EFFECT OF TEMPERATURE ON RUNNING TORQUE

Bearing 21

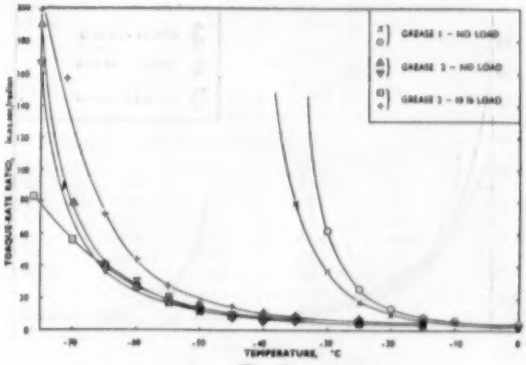

FIG 7

EFFECT OF TEMPERATURE ON RUNNING TORQUE

Bearing 35

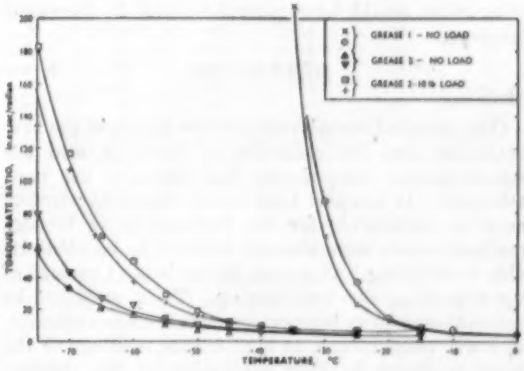

FIG 8

EFFECT OF TEMPERATURE ON RUNNING TORQUE

Bearing 37

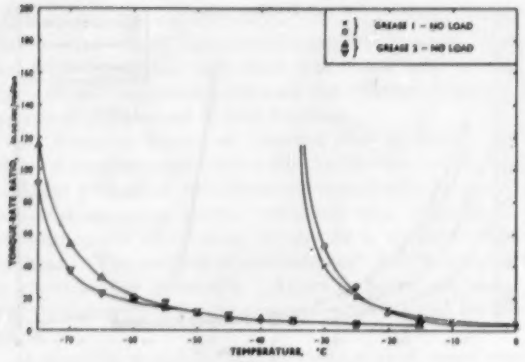

FIG 9

EFFECT OF TEMPERATURE ON RUNNING TORQUE
Bearing 43

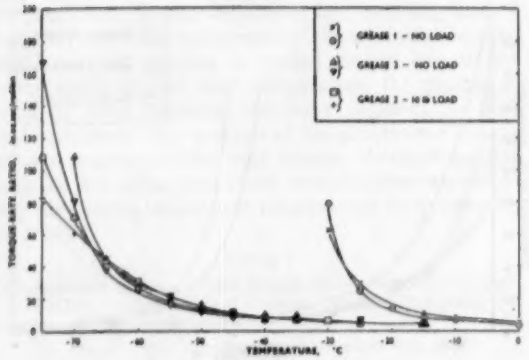

FIG 10

EFFECT OF TEMPERATURE ON RUNNING TORQUE
Bearing 47

istic which would be expected to lead to increased skidding.

DISCUSSION

Skidding

One principal consideration in the design of the new apparatus was the reduction of skidding, and the results quoted have shown that this aim has been achieved. It has also been found that other factors may be responsible for the fluctuations in torque-readings which were thought initially to be attributable to skidding. One such factor is the presence of tight spots in the test bearing. These may not be detectable at room temperature but become apparent, at lower temperature, as high torque readings at the same positions in each revolution of the spindle. Although this effect is undesirable, it does not render the bearing entirely unsuitable for tests under no applied load if there is only one such tight spot, when it is usually possible to obtain a steady reading during part of the revolution. Should there be more than one such spot, or should measurements be made under dead load when the torque variations are magnified by the "flywheel" (inertia) effect of the weights, such bearings must be rejected.

When testing certain types of greases, fluctuations of torque reading may occur in normally satisfactory bearings and cannot be eliminated by loading the bearing even with loads up to about 1000 lb. Under such thrust loads it was thought unlikely that skidding of the balls could occur, and one such system was therefore examined in a cold chamber. It was observed that even when the torque was fluctuating the movement of the cage relative to the inner race was the same as with steady torque readings at room temperature, indicating that "skidding" was not occurring. This particular phenomenon is normally found with greases based on oils which show "crystallization" Pour Points rather than "viscosity" Pour Points, for example, waxy oil or synthetic oils showing "Setting Points." Non-fluctuating torques are found with these materials down to temperatures somewhat below the Pour (or Setting) Point of the base fluid, but the temperature at which fluctuating torques commence does not appear to bear a direct relationship to this Pour or Setting Point. It is probable that the fluctuations in torque result from the intermittent resistance to the rolling of the balls caused by the presence of small areas of hardened grease on the tracks.

Interpretation of Results

It has been stated that the times for rotation, at constant shear stress, with a given grease may vary considerably when using different test bearings in both the British DTD and "Shell" apparatuses, thus leading to an apparent variation in the assessment of the low-temperature suitability of a grease.

Considerable variation in the magnitude of the results has also been obtained with different bearings when used for the determination of running torques on a grease in the "Shell" apparatus. This variation has been observed particularly when bearings made by different manufacturers have been examined. An extreme case of such variation is illustrated in Fig 11, where two bearings of different manufacture were used to examine Grease 3. Hence, if, as is sometimes done, the minimum service temperature is defined as that

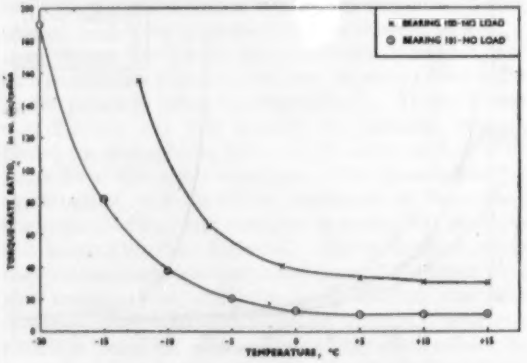

FIG 11

EFFECT OF TEMPERATURE ON RUNNING TORQUE
Grease 3

TABLE III

Minimum Operating Temperature (° C)—Tests at 2 r.p.m.

Bearing No.	Method of assessment							
	(*a*)				(*b*)			
	Test 1		Test 2		Test 1		Test 2	
	Operator X	Operator Y	Operator X	Operator Y	Operator X	Operator Y	Operator X	Operator Y
	Grease 1—without additional load							
3	−27	−27	−28	−28	−19·5	−19	−19·5	−19
19	−23	−23	−23	−22	−19	−17	−19	−15
21	−23	−23	−24	−23	−16	−15	−16	−15
35	−26	−26	−29	−29	−18·5	−18	−18·5	−19
37	−24	−24	−26·5	−26	−18·5	−20	−19	−21
43	−26·5	−25	−28	−28	−19	−19·5	−20	−18
47	−25·5	−26	−26	−26	−19	−19	−20	−20
Mean	−25	−25	−26·4	−25·5	−18·5	−18·2	−18·9	−18·1
Overall mean	−25·5				−18·4			
Overall range	−22 to −29				−15 to −21			
	Grease 2—without additional load							
3	−70	−69	−71	−71	< −75	−60	−75	−55
19	−57	−57	−62	−63	−60	−51	−60	−53
21	−61·5	−61	−67	−68	−59	−58	−59	−57
35	−61	−60	−63	−61	−61	−50	−63	−50
37	−66·5	−65	−69	−69	−60	−62·5	−63·5	−60
43	−64	−62	−68	−67	−58	−55	−61	−59
47	−61·5	−61	−62·5	−62	−60	−60	−60	−59
Mean	−61·9	−61	−65·2	−65	−59·7	−56·1	−61·1	−58·3
Overall mean (excluding bearing 3)	−63·3				−58·3			
Overall range (excluding bearing 3)	−57 to −69				−50 to −63·5			
	Grease 2—with 10 lb load							
3	−57	−56	−57	−59	−57	−57	−57	−54
19	−53	−53·5	−58	−58·5	−50·5	−51	−55	−51·5
21	−55	−54·5	−55	−55	−50	−49·5	−50	−52
35	−56	−56	−62·5	−61	−55	−57·5	−58·5	−54
37	−55	−55·5	−57	−58	−51	−52·5	−51	−53
43	—	—	—	—	—	—	—	—
47	−59·5	−60	−60	−60	−57·5	−56	−57	−57
Mean	−55·9	−55·9	−58·2	−58·6	−53·5	−53·9	−54·7	−53·6
Overall mean	−57·2				−53·9			
Overall range	−53 to −62·5				−49·5 to −58·5			

	Grease 3—without additional load			
	Operator X	Operator Y	Operator X	Operator Y
100	Approx +12	Approx +12	−9	−9
101	−8	−9	−8	−9

at which the torque function reaches a given value (Method (*a*) of Table III), the assessment is dependent on the test bearing used and poor repeatability results. As an example, if a torque-rate ratio of 30 in oz. sec/radian * be selected as the arbitrary value, then the data of Fig 11 give minimum service temperatures of +10° to +15° C for bearing 100 and −8° C for bearing 101, a wide variation.

* This has been chosen following an examination of greases meeting various specifications and corresponds approximately to a low-temperature torque time in the DTD apparatus of 5 sec at the minimum service temperature.

An empirical method of assessment (Method (*b*) of Table III), which might be expected to be less dependent on bearing geometry than the method (*a*), is based on the rate at which the torque function increases with decrease in temperature. The minimum service temperature is taken as that point on the torque-function–temperature curve at which the torque has tripled over an interval of 20° C. When applied to the data of Fig 11 Method (*b*) gives minimum service temperatures of —9° C for bearing 100 and —8° C for bearing 101, representing a considerable improvement in repeatability.

A possible flaw in Method (*b*), however, is that the assessment will be more dependent on the shape of the curve drawn through the experimental results than with Method (*a*). Some indication of this effect may be obtained from the data tabulated in Table III, where the results given in Figs 5–11 have been analysed by two operators, X and Y, using Methods (*a*) and (*b*). There is an indication, especially in the case of the tests without added load, that Operator X constructs more " idealized " curves than Operator Y.

It is also apparent from the results with this series of bearings, which were all supplied by one manufacturer, that Methods (*a*) and (*b*) give assessments showing approximately the same degree of variation. If therefore it is possible to select test bearings either by taking supplies from one source or by measuring some particular characteristic of the bearings, then the use of Method (*b*) for assessing low-temperature characteristics will offer no advantage in precision over Method (*a*). With unselected bearings there may be an advantage in precision by using Method (*b*). Some form of co-operative work is, however, desirable to evaluate fully both the " Shell " apparatus and the proposed method of assessing minimum operating temperatures, and such work is under consideration by the IP Mechanical Tests of Greases Panel.

ACKNOWLEDGMENT

The author wishes to thank the directors of " Shell " Research Ltd. for permission to publish this paper.

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APPENDIX

The Determination of Running Torques Using the " Shell " Apparatus

The test bearing shall be cleaned by the use of suitable solvents and then weighed to within 0·1 g. A metal collar which is an easy fit on the outside diameter of the bearing shall be rested on a flat glass or metal plate, and the bottom of the space so formed shall be covered with the test grease to a depth of about $\frac{1}{4}$ inch. The bore of the test bearing shall be closed by means of a cork and the bearing pushed into the collar while the inner race is rotated slowly by hand, thus distributing the grease over the surfaces of the bearing. The test bearing shall be removed from the collar, the external surfaces of the bearing wiped free of grease, and the grease charge adjusted to $3 \pm 0{\cdot}1$ g.

The bearing shall be mounted in the test apparatus and secured in position by the locking ring on the outer race and the face plate and nut on the inner race. Care shall be taken during mounting to ensure that the thermocouples are correctly located, namely one in contact with the face of the outer race of the bearing and the other floating in the air gap above the cage but not in contact with any metal. The liquid-tight cover shall be screwed in position after smearing the rubber sealing ring with a suitable lubricant. The test spindle shall be rotated one hundred times in each direction and immersed in the alcohol cold bath at room temperature. The pulley shall be mounted on the spindle and the thread from the constant-speed drive wound round the pulley at least four times in such a way that the turns do not overlap. Any desired load is then applied to the spindle by mounting annular-shaped weights above the pulley.

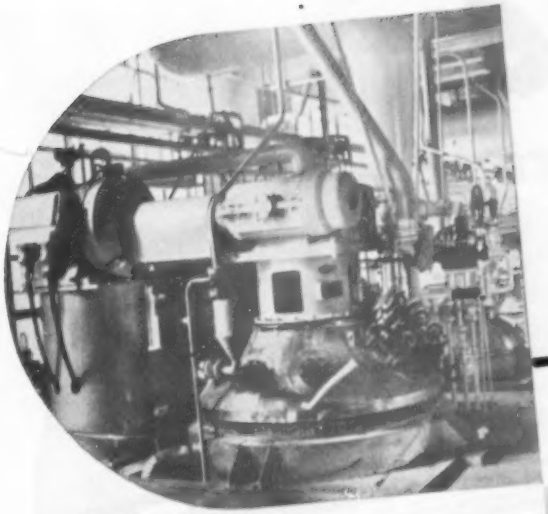
Powdered solid carbon dioxide shall be added to the alcohol bath, which is agitated by bubbling dry air, and the temperature of the bath, measured by a suitable thermometer immersed therein, reduced to a level approx 2° C below that at which the first measurement is to be made. This results in a relatively slow rate of cooling of the bearing so that a measurement may be made as soon as the bearing reaches the desired temperature without the necessity of soaking for prolonged periods. The measurement shall be made by starting the constant speed motor and recording the steady reading of the spring balance after two revolutions of the spindle. The motor shall then be reversed and the spindle slowly rotated by hand in the reverse direction until the original position is approximately reached. The motor shall be switched off, the bath temperature reduced to about 2° C below the next test temperature and the measurements repeated. Measurements shall be repeated at successively reduced temperatures until the desired temperature range has been covered or until the torque requirement becomes excessive, *i.e.* exceeds the range of available spring balances or a previously selected value.

Calculation of Results

The results shall be reported as torque-rate ratios at the test speed and shall be calculated as follows:

$$\text{Torque-rate ratio} = \frac{2{\cdot}39Ld}{n}$$

where L is the spring balance reading; d is the effective pulley diameter on the spindle; n is the spindle speed in r.p.m.; L and d being in ounces and inches respectively, the torque-rate ratio is reported as " inch ounce sec/radian."



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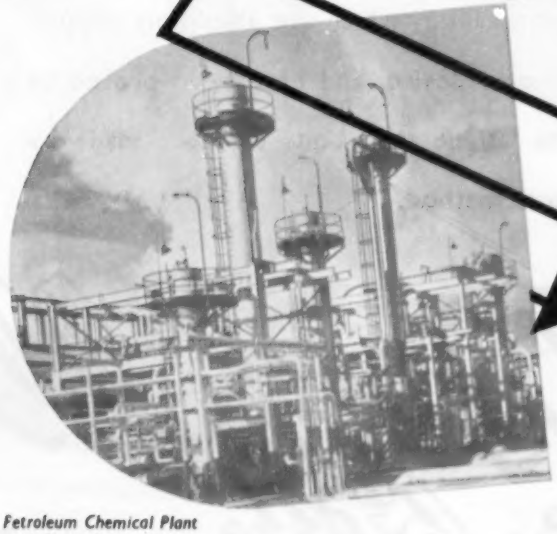

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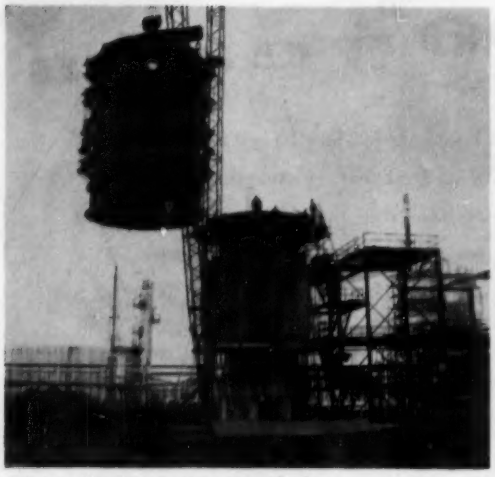
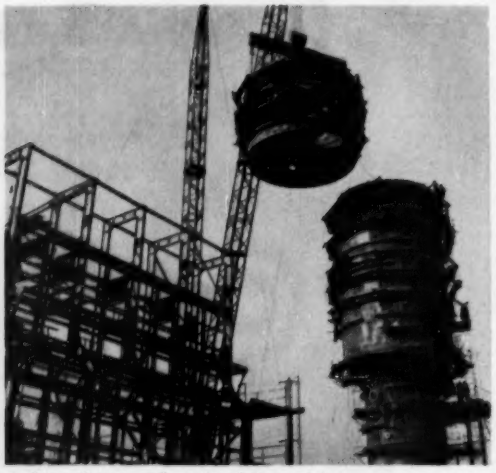
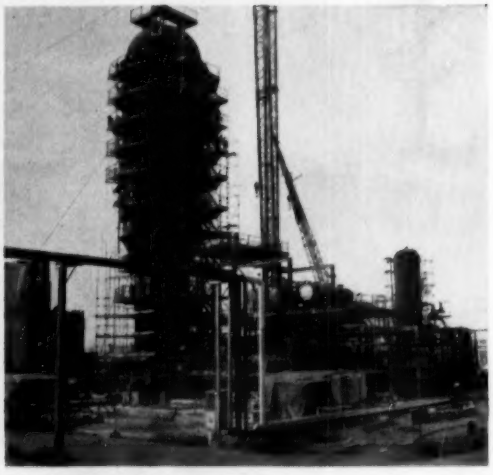


Putting the first section on to the base



Lifting the second section into position

Preparing to raise the domed head



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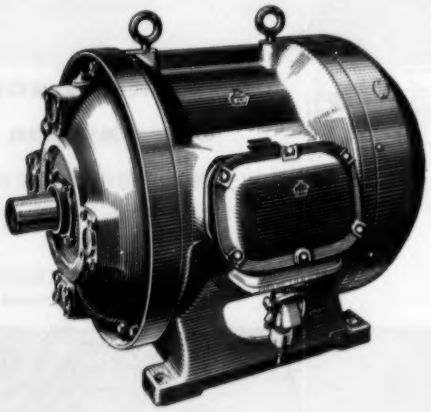

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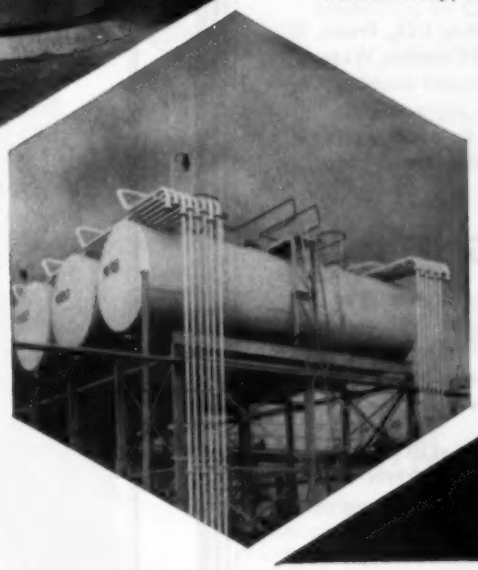

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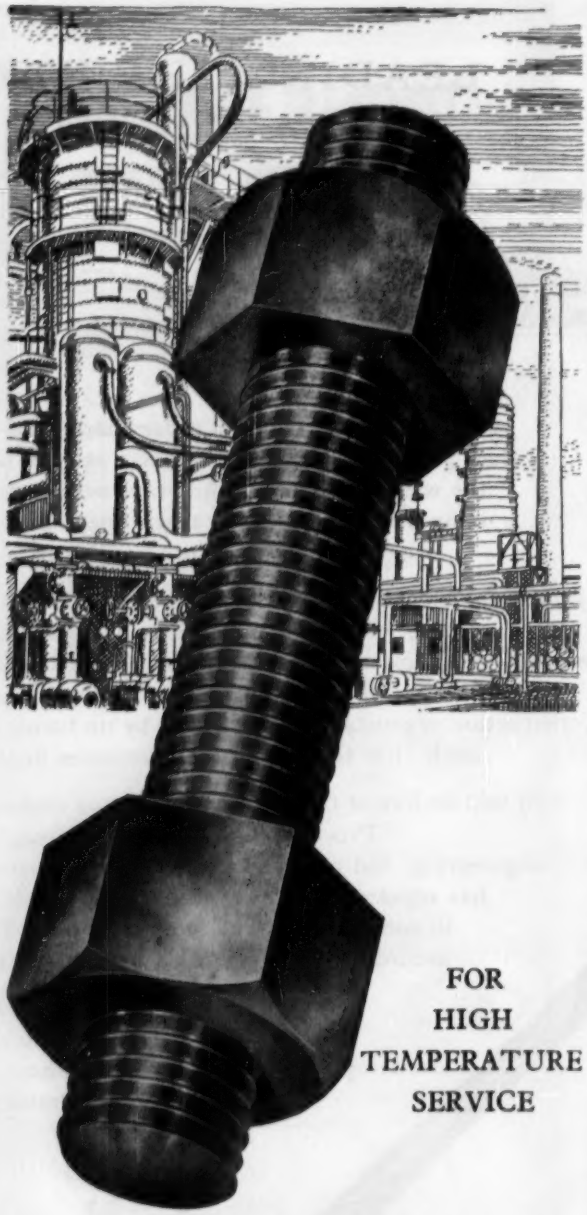

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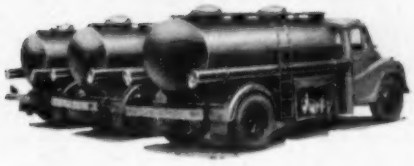

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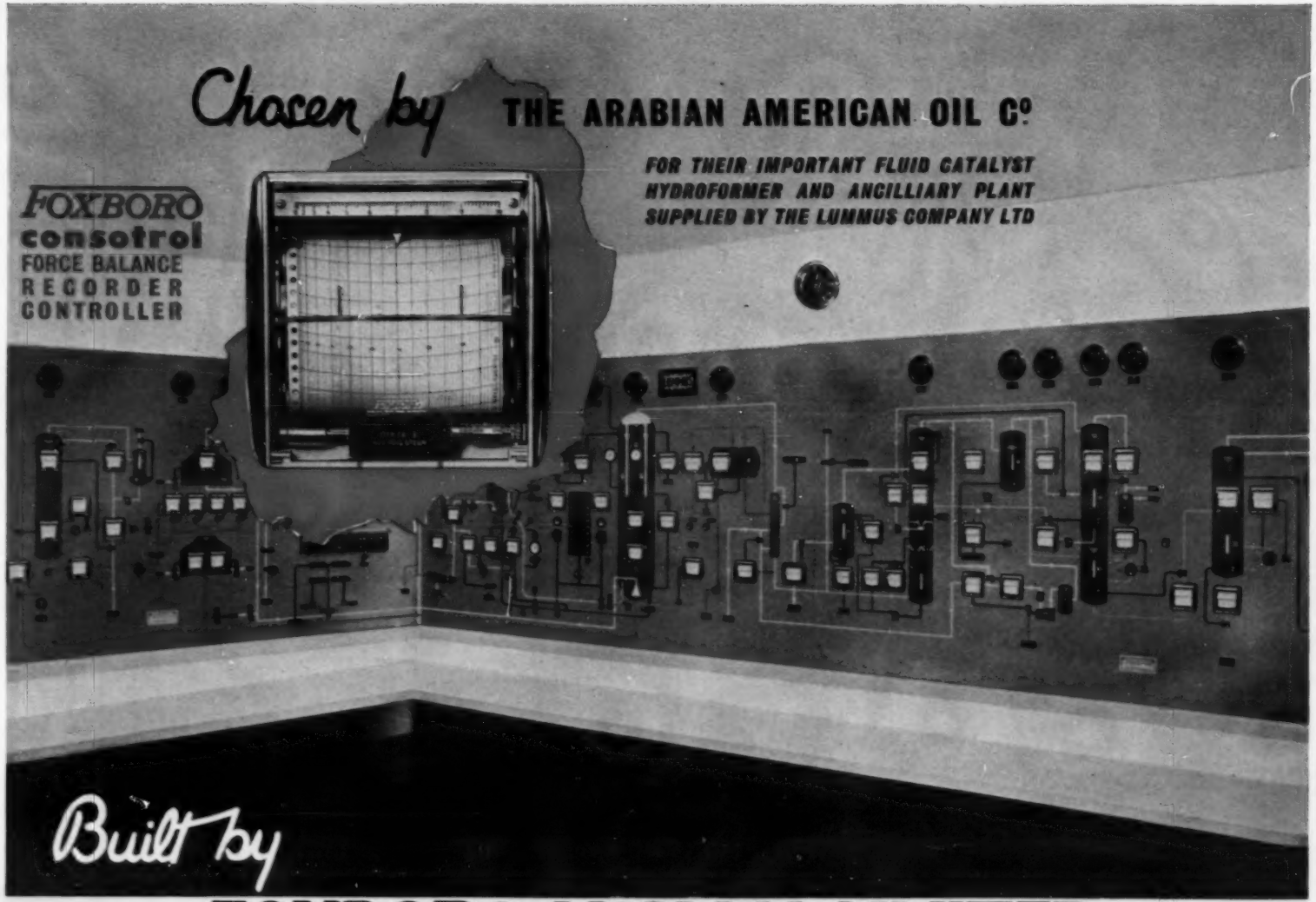

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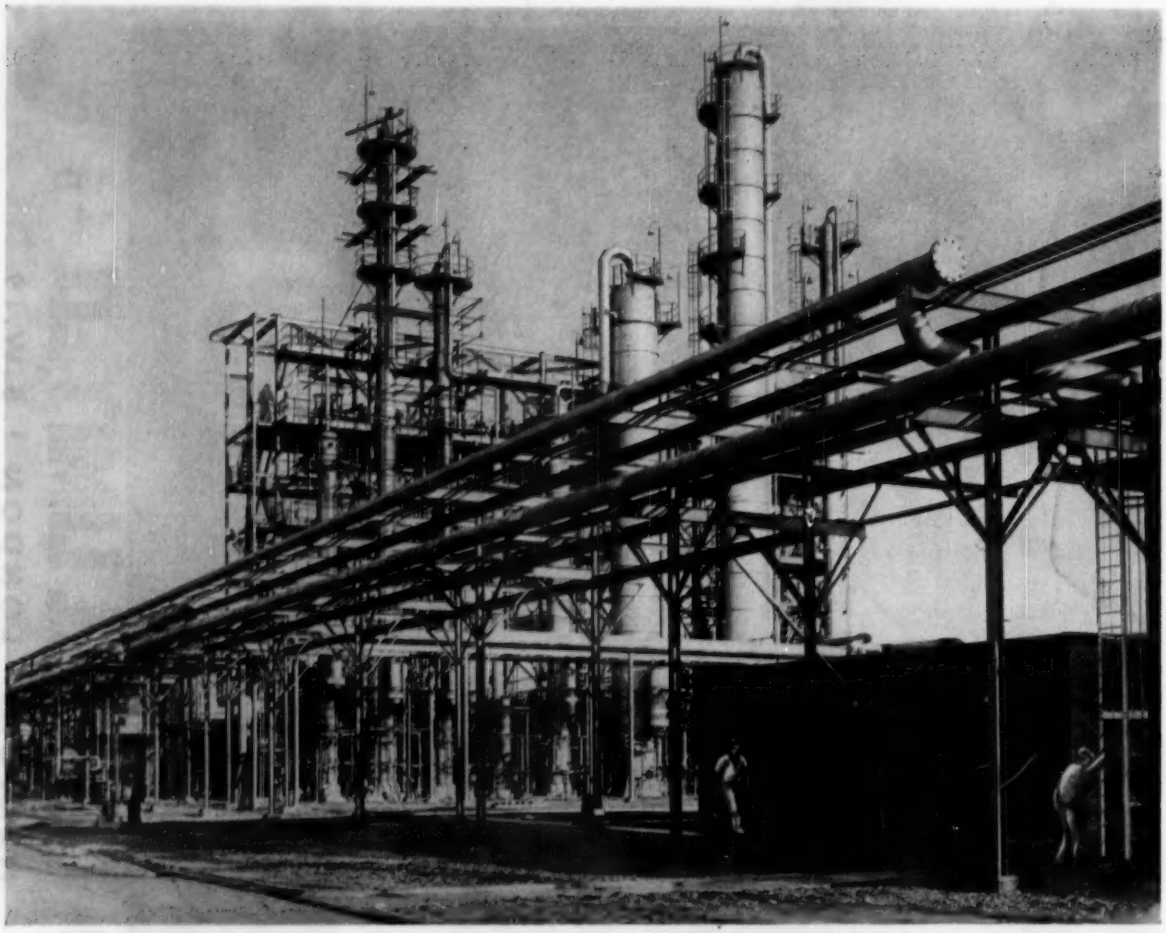

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